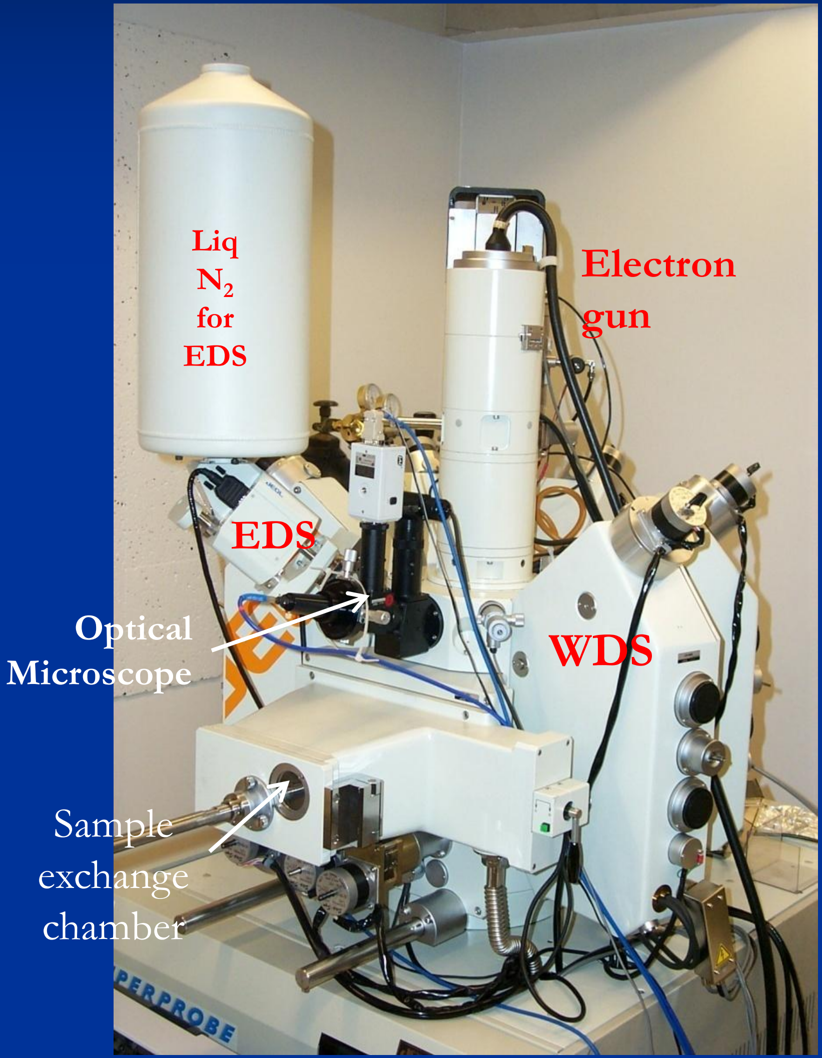
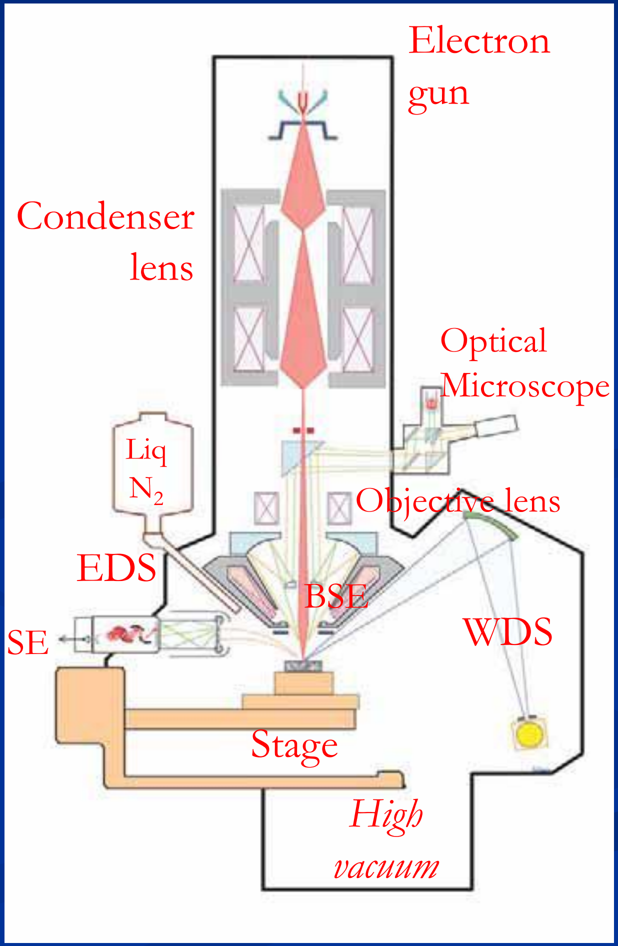
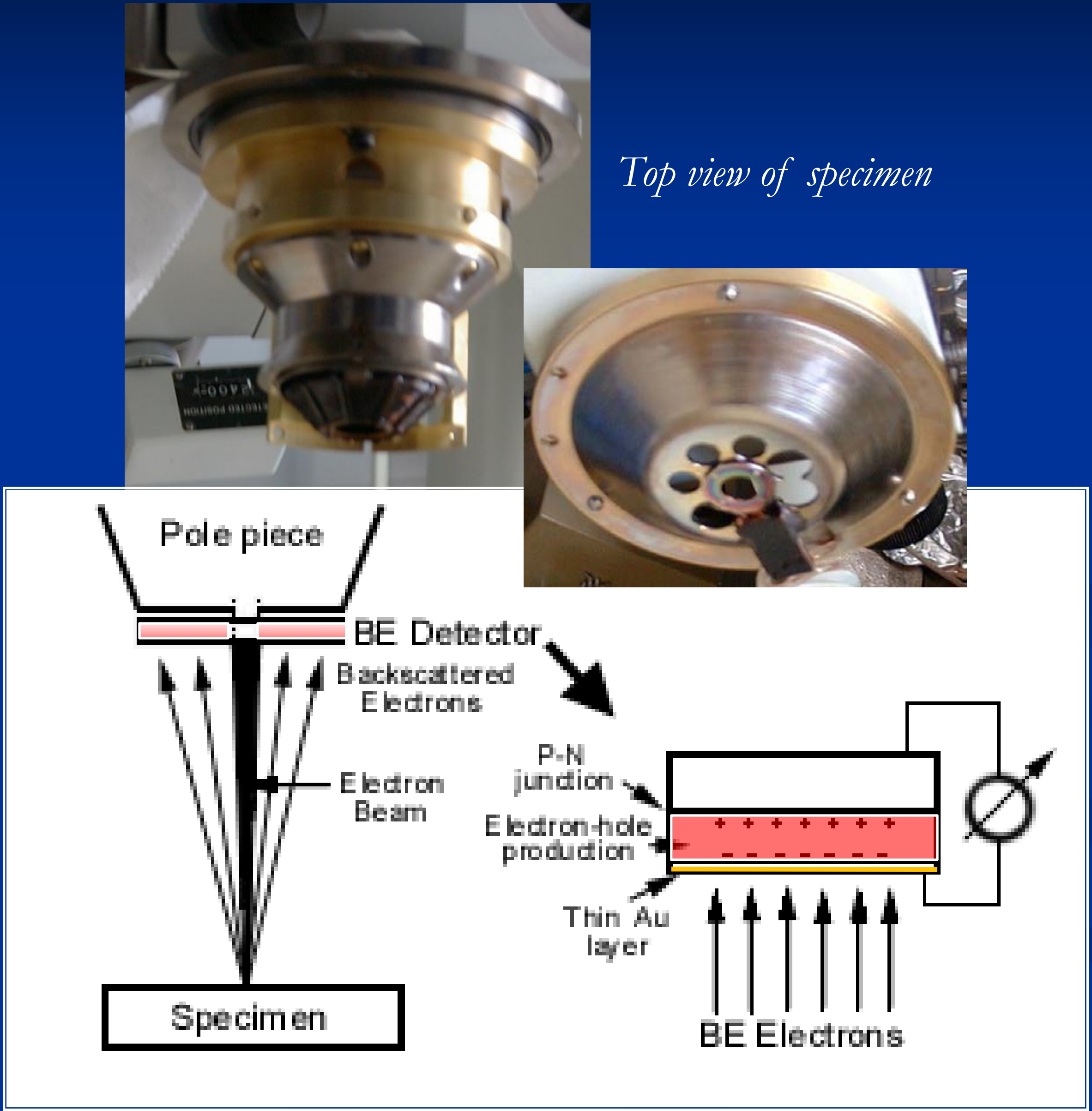
JEOL JXA-8200 Superprobe





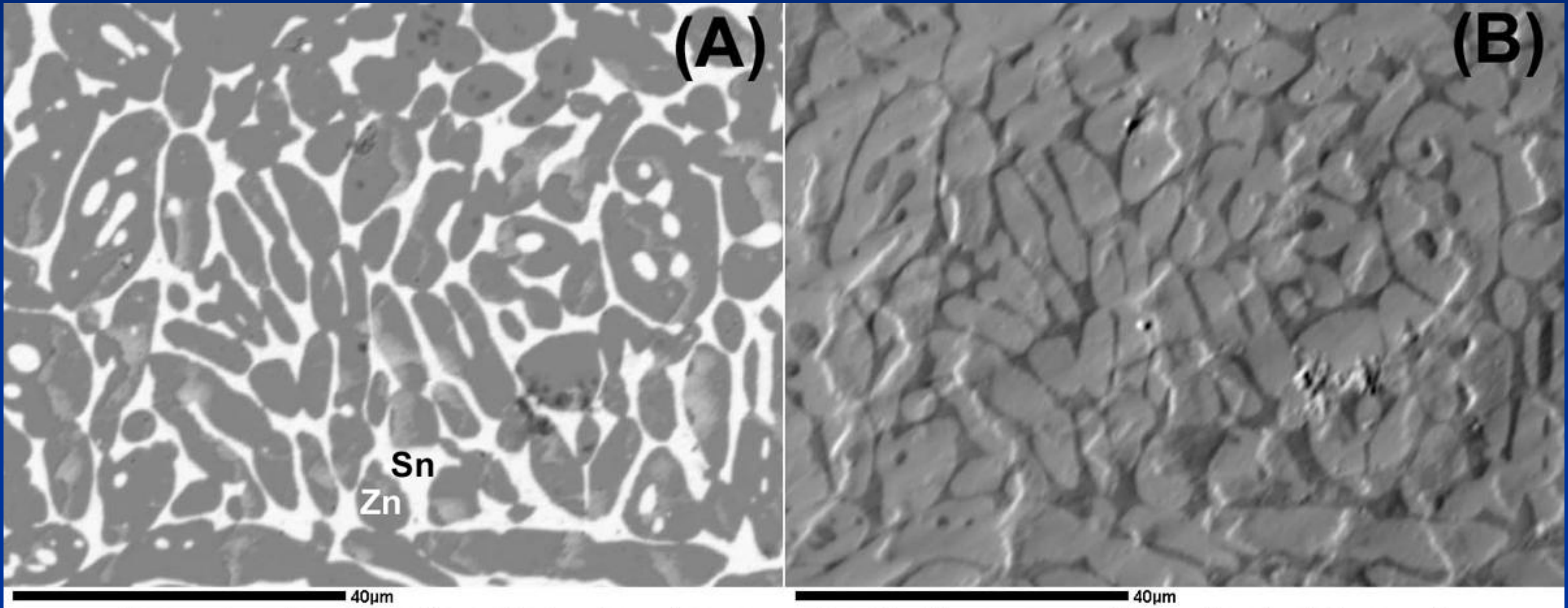
Perpendicular geometry

Back-scattered electron detector



Top view of specimen

Compositional and topographic imaging with BSE



Scanning backscattered electron images of a Zn-Sn composite collected through a solid-state diode detector in (A) A+B, or compositional mode; (B) A-B, or topographic mode

A+B
Compositional mode

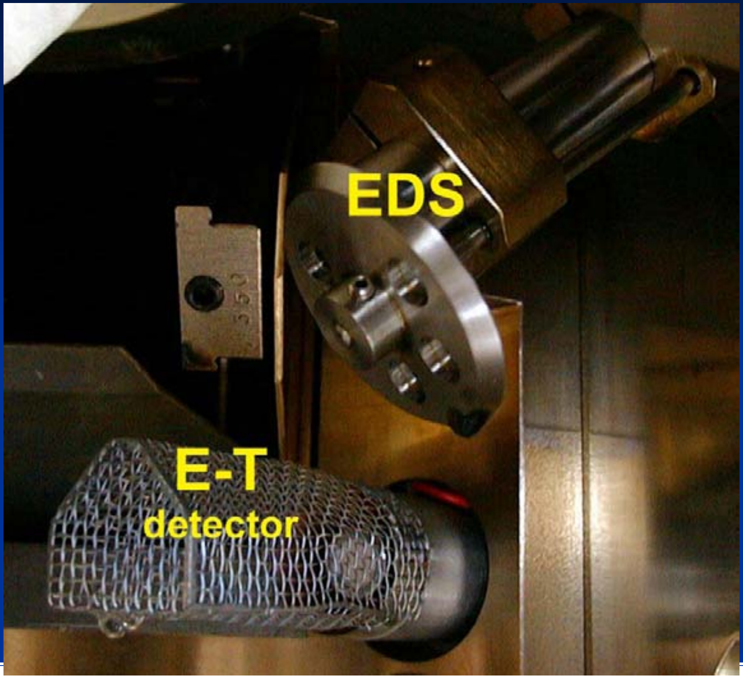
A-B
Topographic mode

Secondary electrons

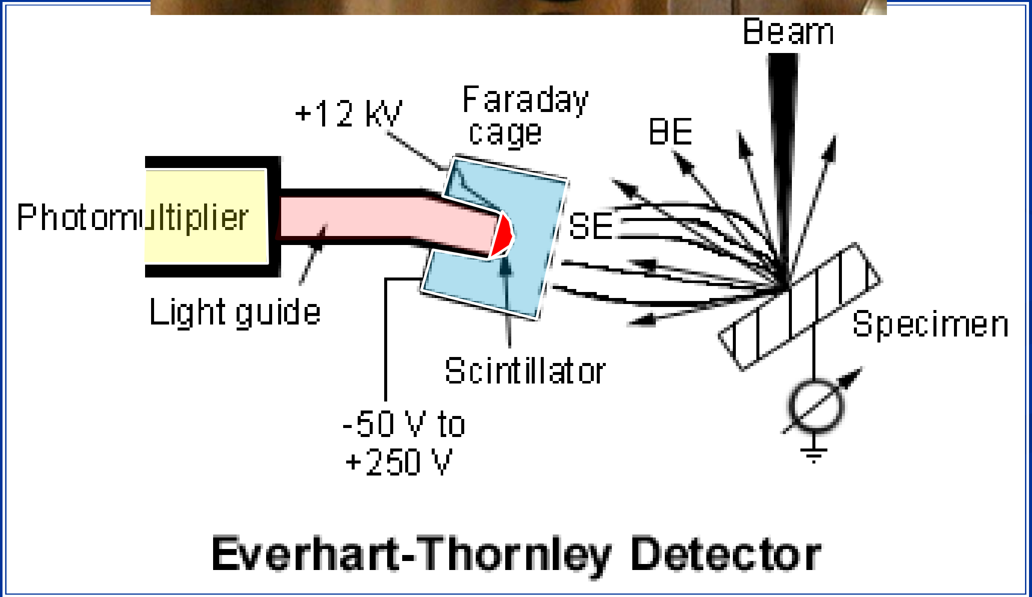
- Specimen electrons mobilized by electron beam through inelastic scattering
- Secondary electrons have lower energy compared to backscattered electrons

(useful in studying surface topography)

Secondary electron detector

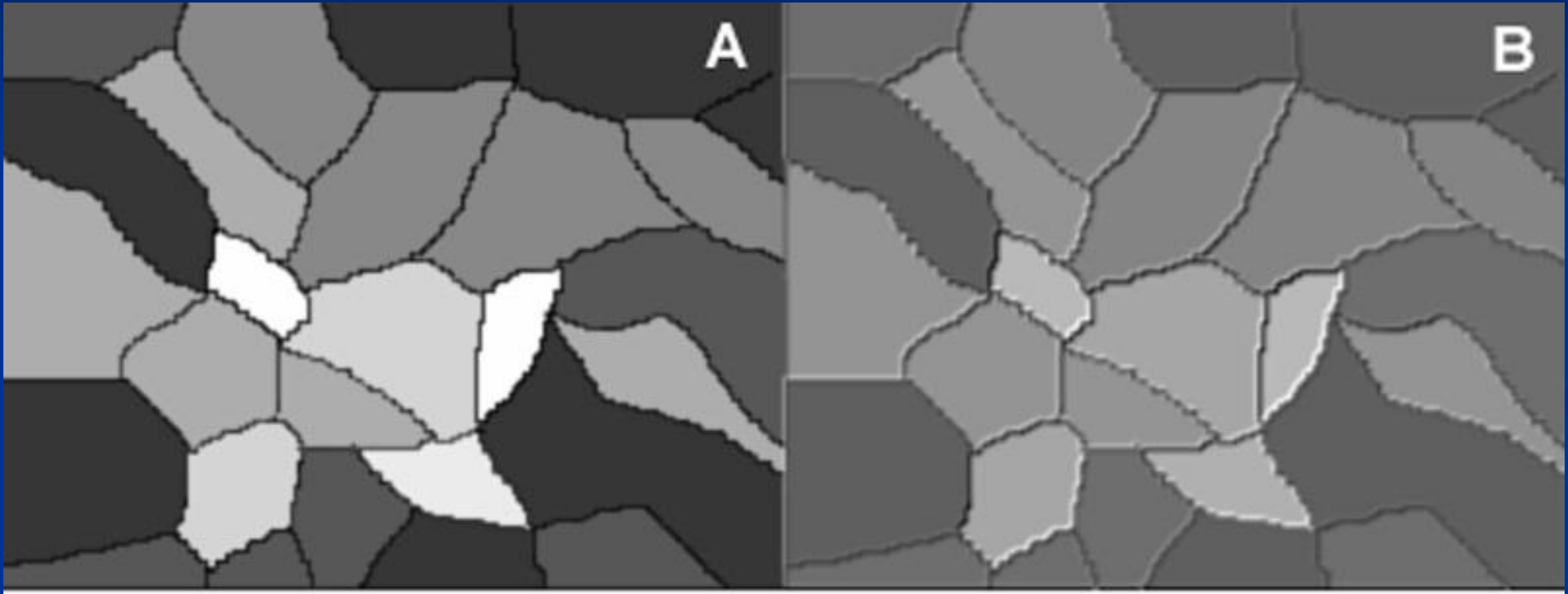


Side view of specimen



Everhart-Thornley Detector

Secondary electron imaging



(A) negatively biased, (B) positively biased E-T detector

-ve Faraday cage bias

less SE

less topographic contrast

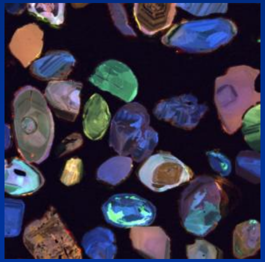
+ve Faraday cage bias

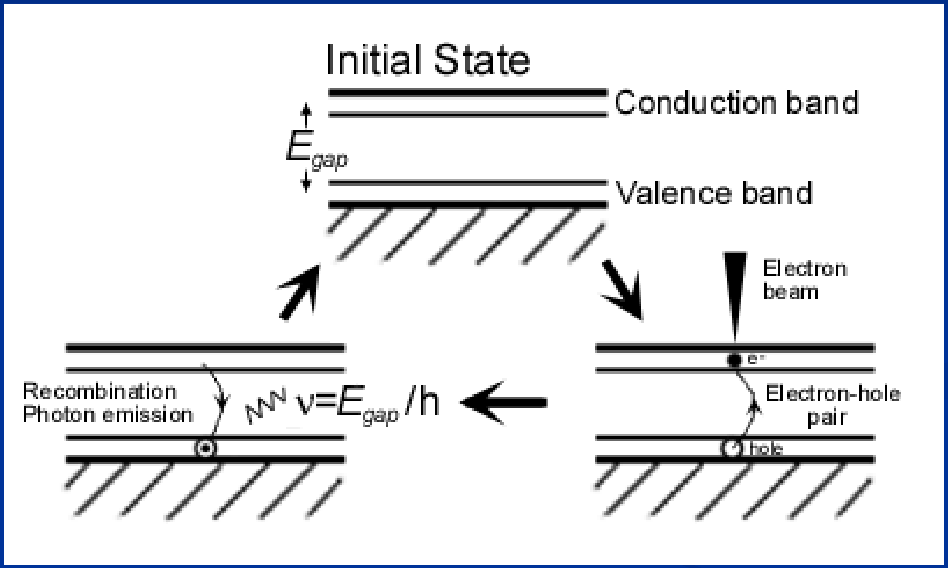
more SE

better topographic contrast

Cathodoluminescence

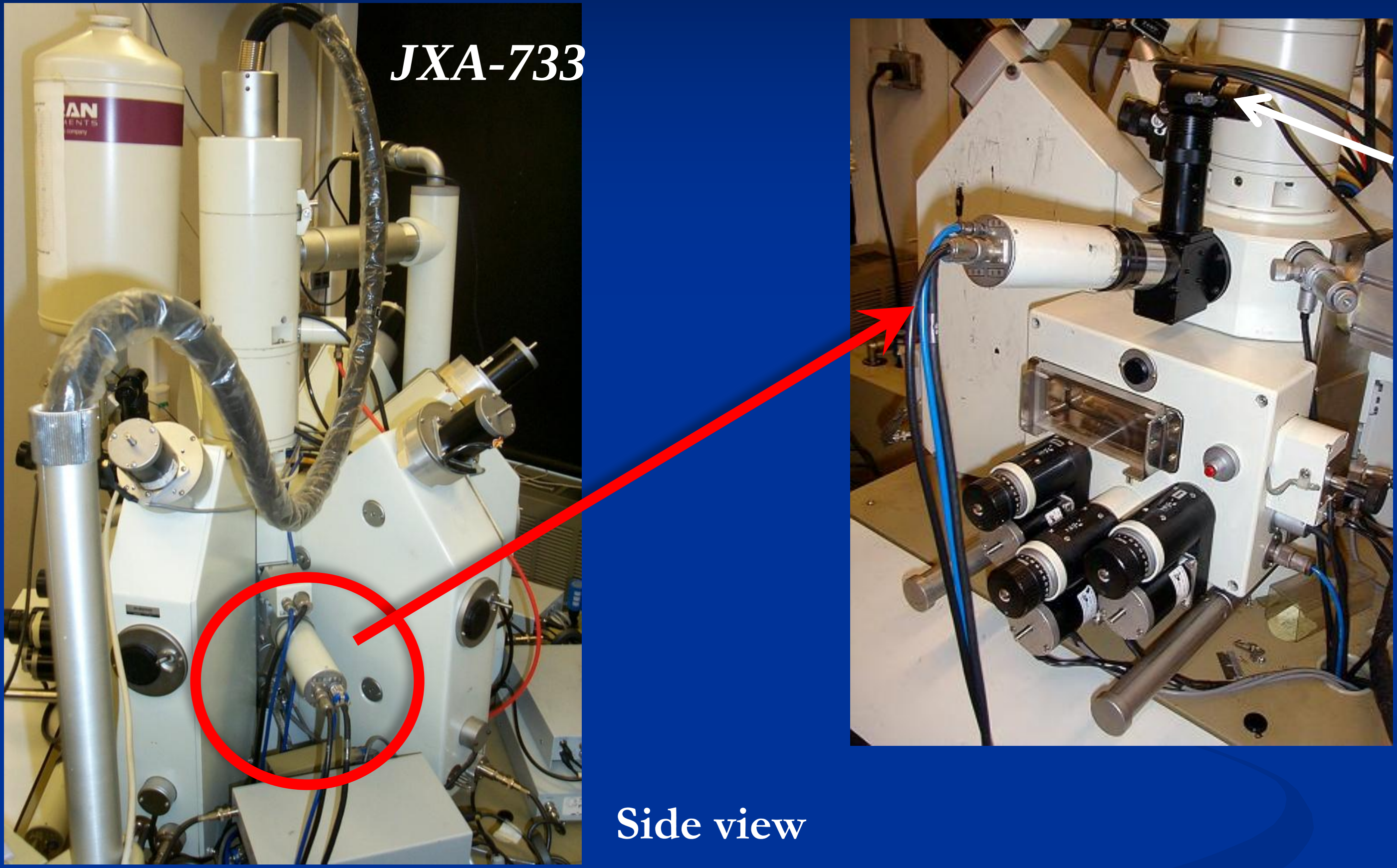
Light generated from sample through electron beam interaction





- *Band gap energy, E_{gap}, is a property of the semiconductor*
- *Trace impurities change E_{gap} by adding additional energy states in the band gap*

Cathodoluminescence detector



Optical
microscope
light
turned off

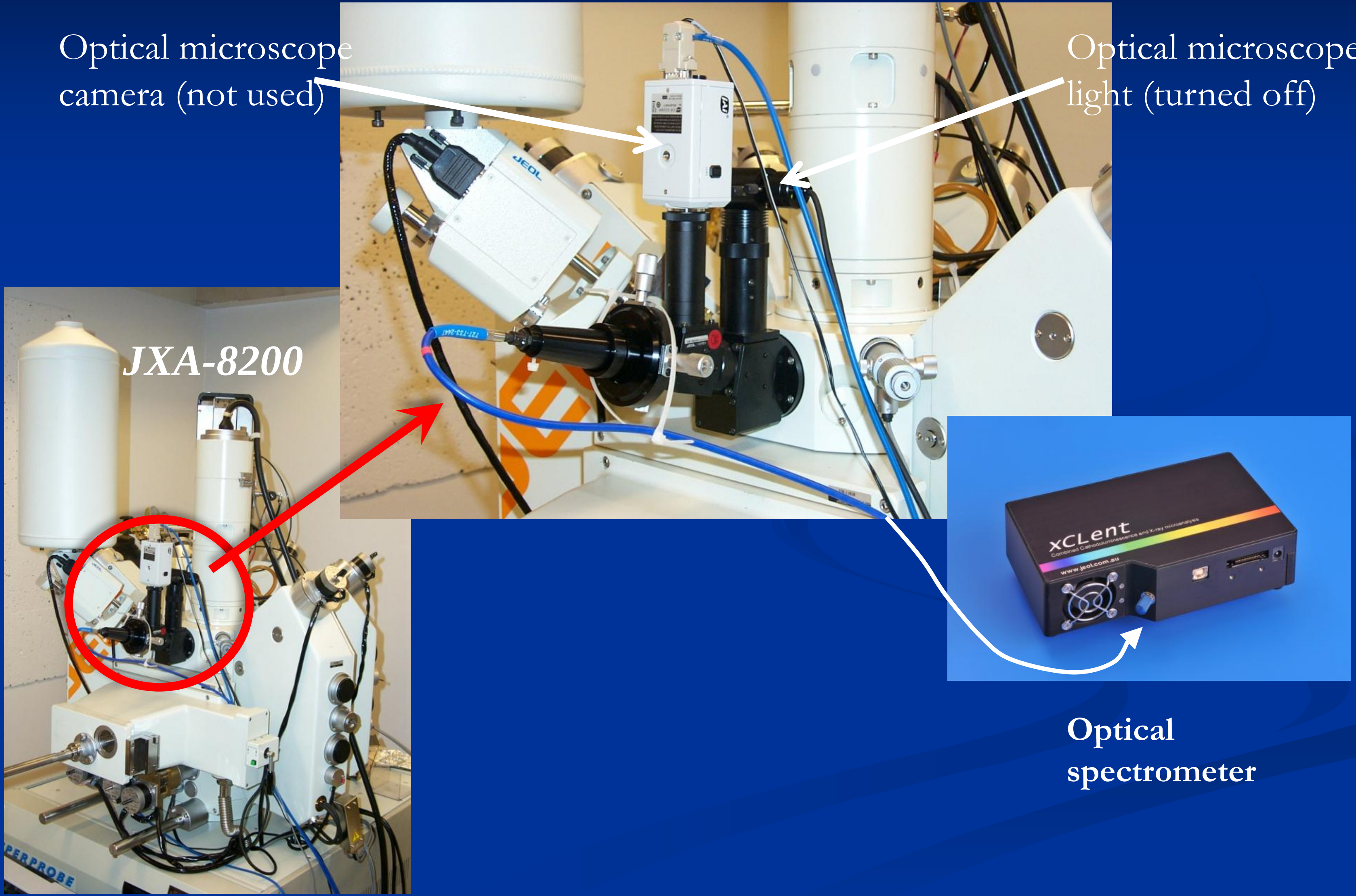
Front view

Side view

Photomultiplier for secondary electron imaging is used as CL detector

Optical arrangement is the same as for the optical microscope

Cathodoluminescence spectrometer

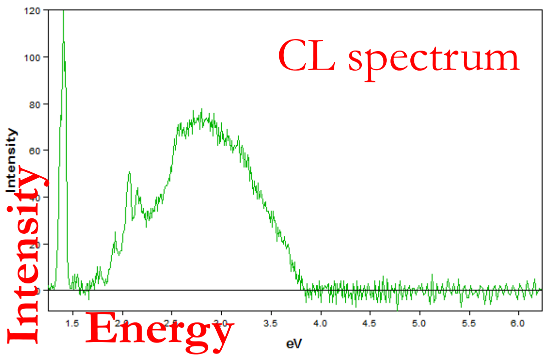


Optical microscope camera (not used)

Optical microscope light (turned off)

Optical spectrometer

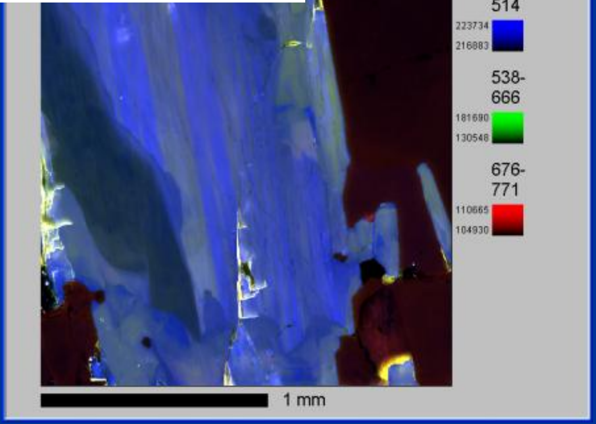
Integrated CL and X-ray spectrometry



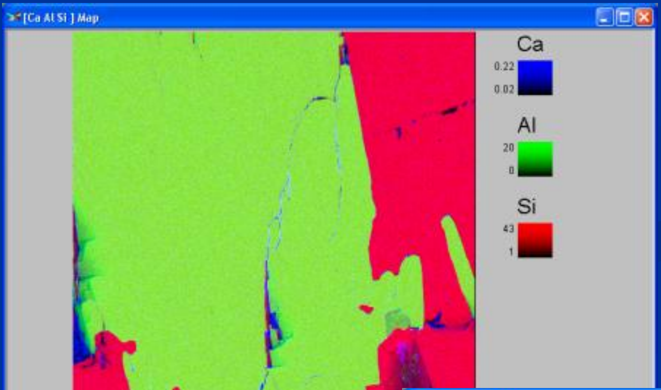
CL spectrum

Intensity

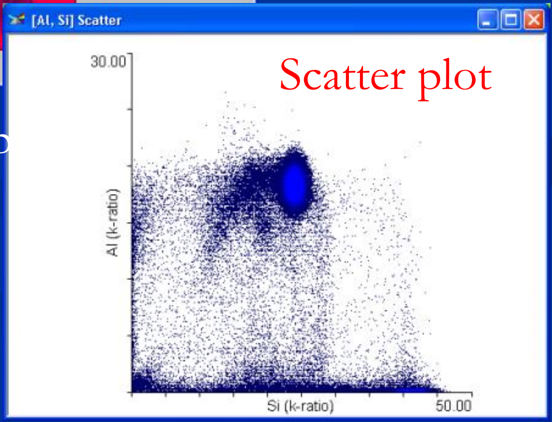
Energy



3 band CL map



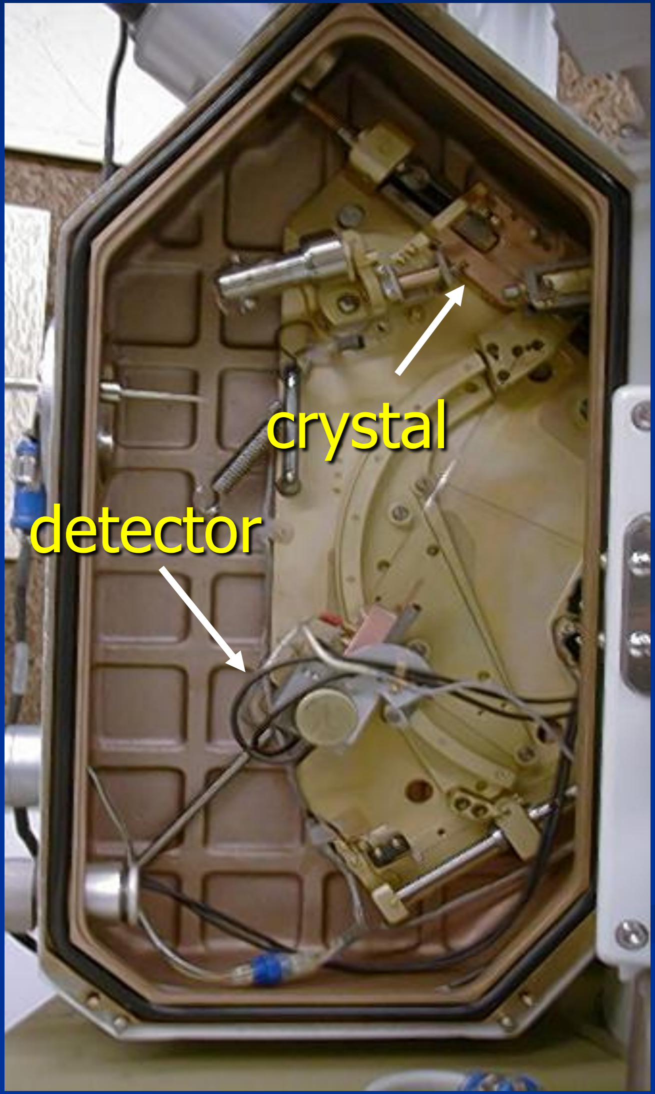
3 element X-ray map



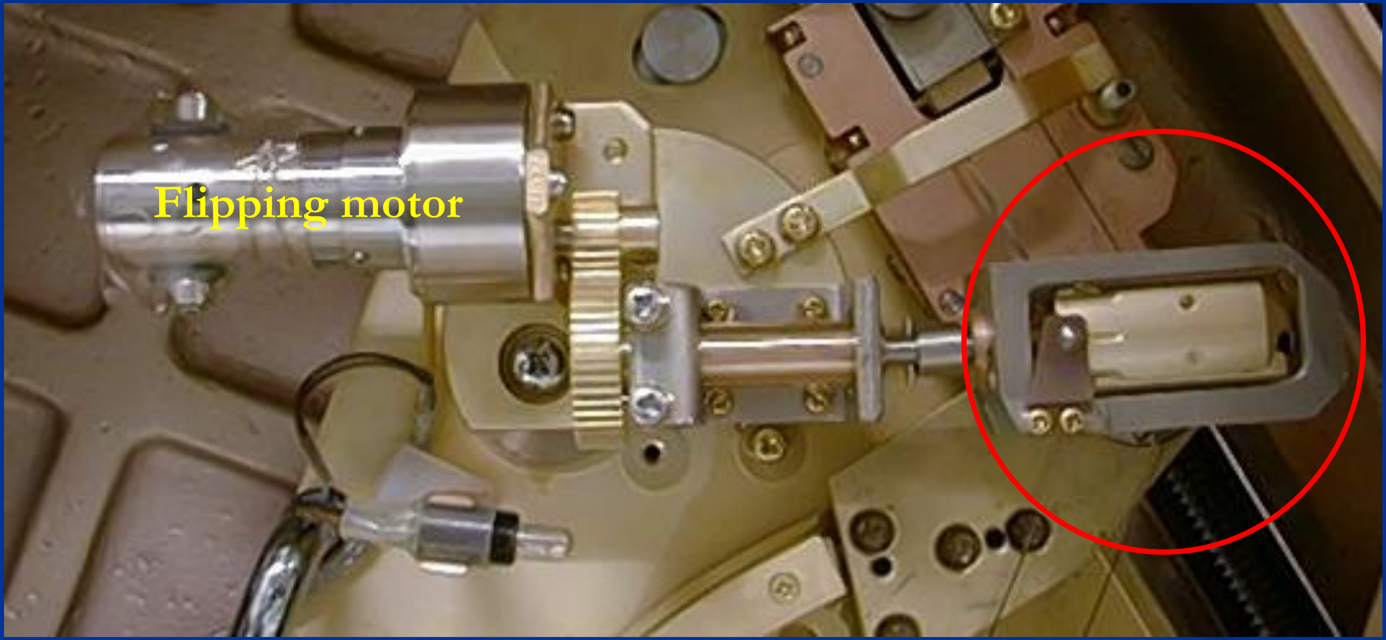
Scatter plot

CL wavelength band may be correlated with an element

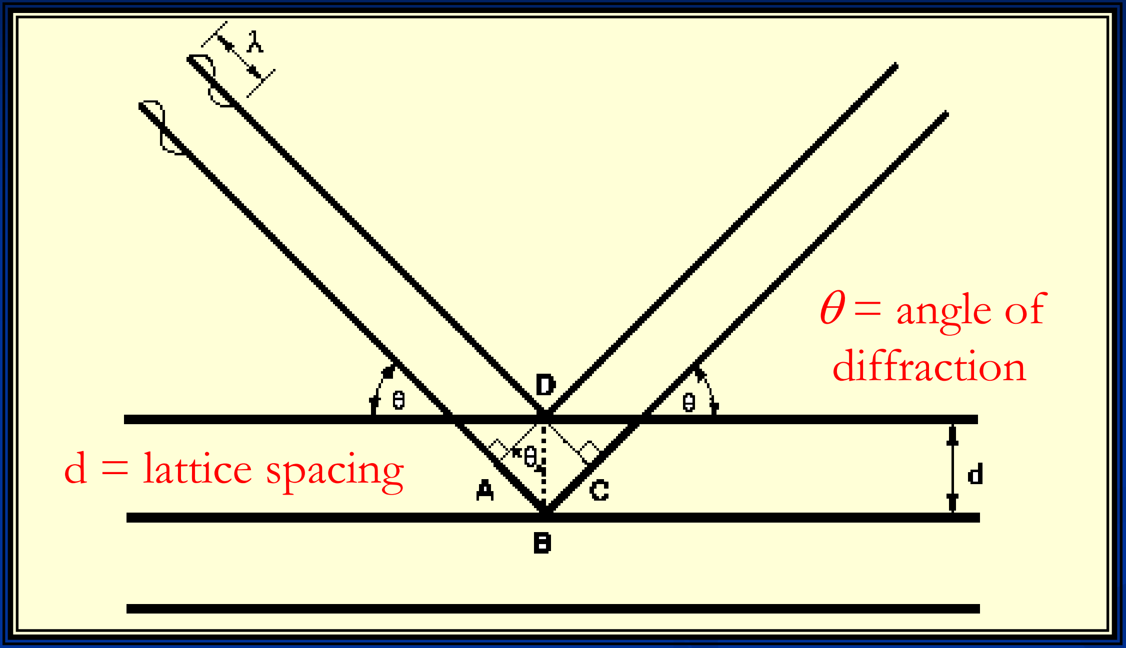
Wavelength Dispersive Spectrometer (WDS)



WDS Analyzing crystal



Bragg's Law



θ = angle of diffraction

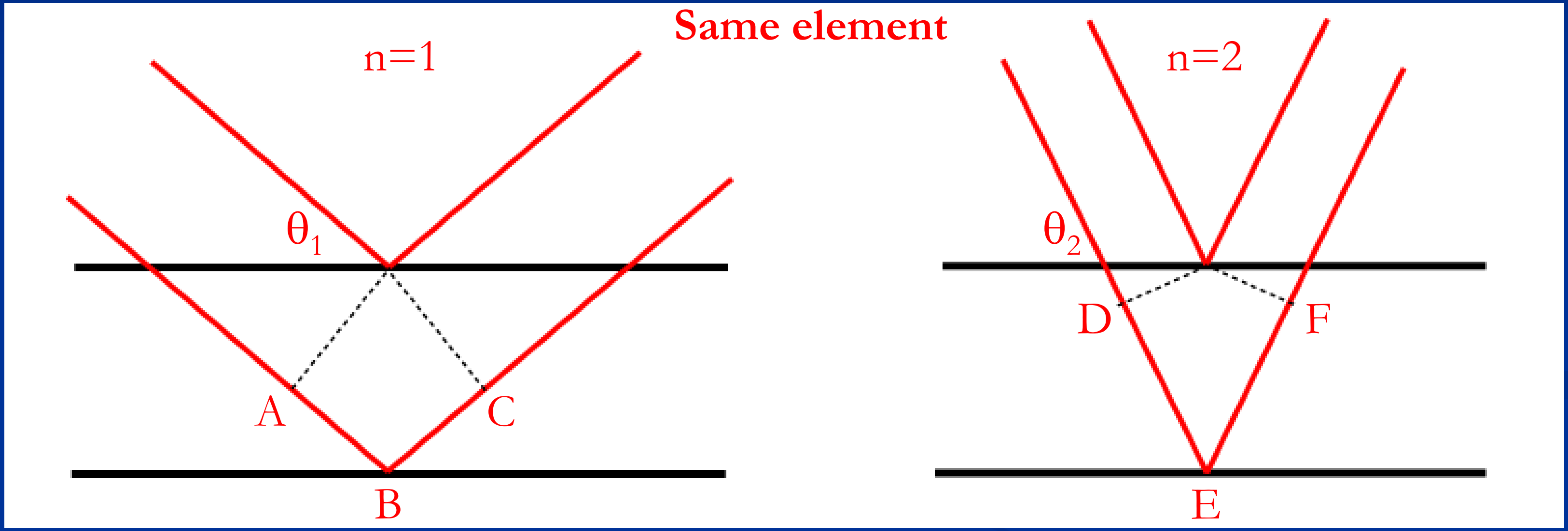
d = lattice spacing

$$\boxed{n\lambda = 2d\sin\theta}$$

= path length ABC

n = order of reflection
(any integer)

First and second order reflections

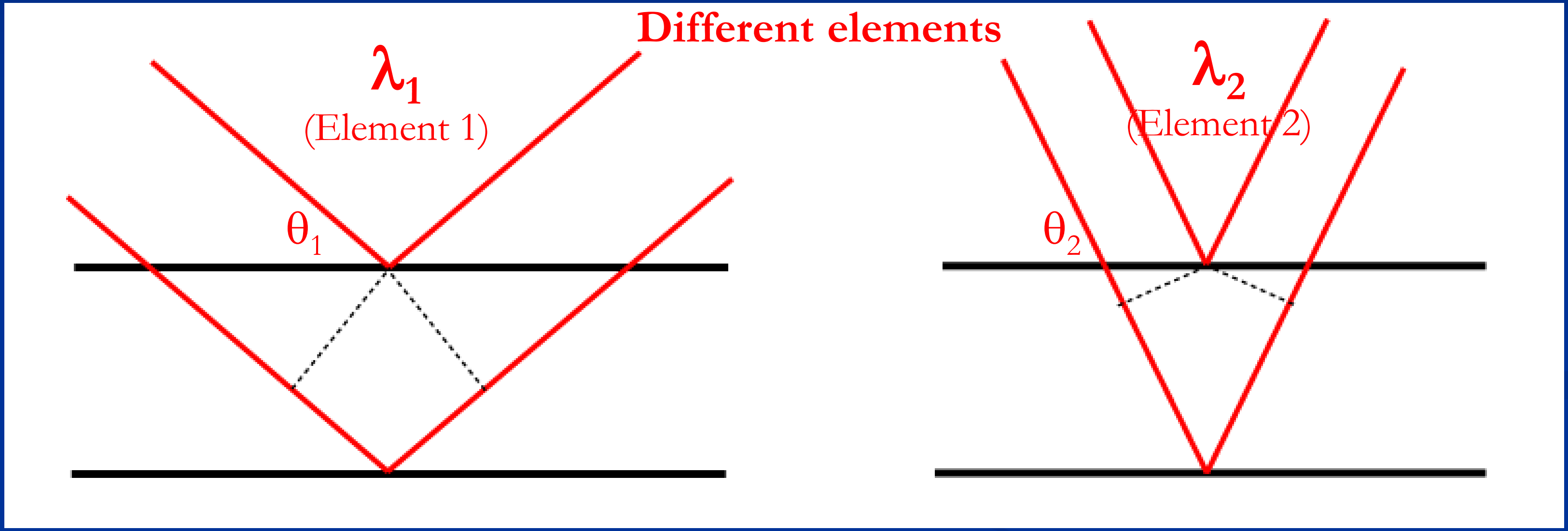


$1\lambda = 2d \sin\theta_1$
=ABC

$2\lambda = 2d \sin\theta_2$
=DEF

path DEF = 2* path ABC

Diffraction angle



$n\lambda_1 = 2d \sin\theta_1$

$n\lambda_2 = 2d \sin\theta_2$

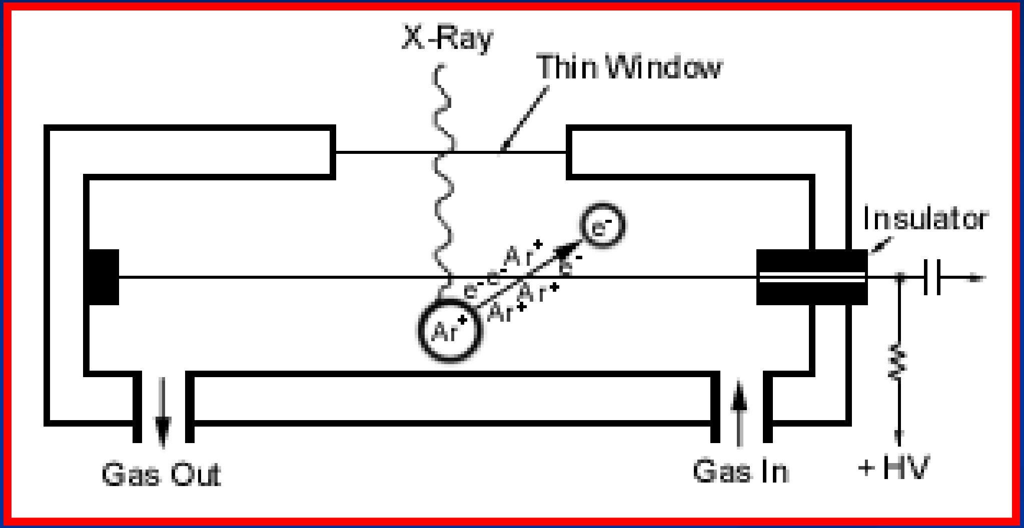
Diffraction angle changes with wavelength being diffracted (for the same order of reflection, n)

WDS Analyzing crystals with different "d" spacings

Name	2d (Å)	Type	Elements usually analyzed
LDEC	98	Ni/C Multi-layer	B-O (Kα), optimized for C analysis
STE	100.4	Pb stearate	B-O (Kα), optimized for C analysis
LDE1	59.8	W/Si Multi-layer	C-F (Kα), optimized for O analysis
TAP	25.8	Thallium acid phthalate	Na-P (Kα); Cu-Zr (Lα); Sm-Au (Mα)
PET	8.742	Pentaerythritol	S-Mn (Kα); Nb-Pm (Lα); Hg-U (Mα)
LIF	4.028	Lithium fluoride	Ti-Rb (Kα); Ba-U (Lα)

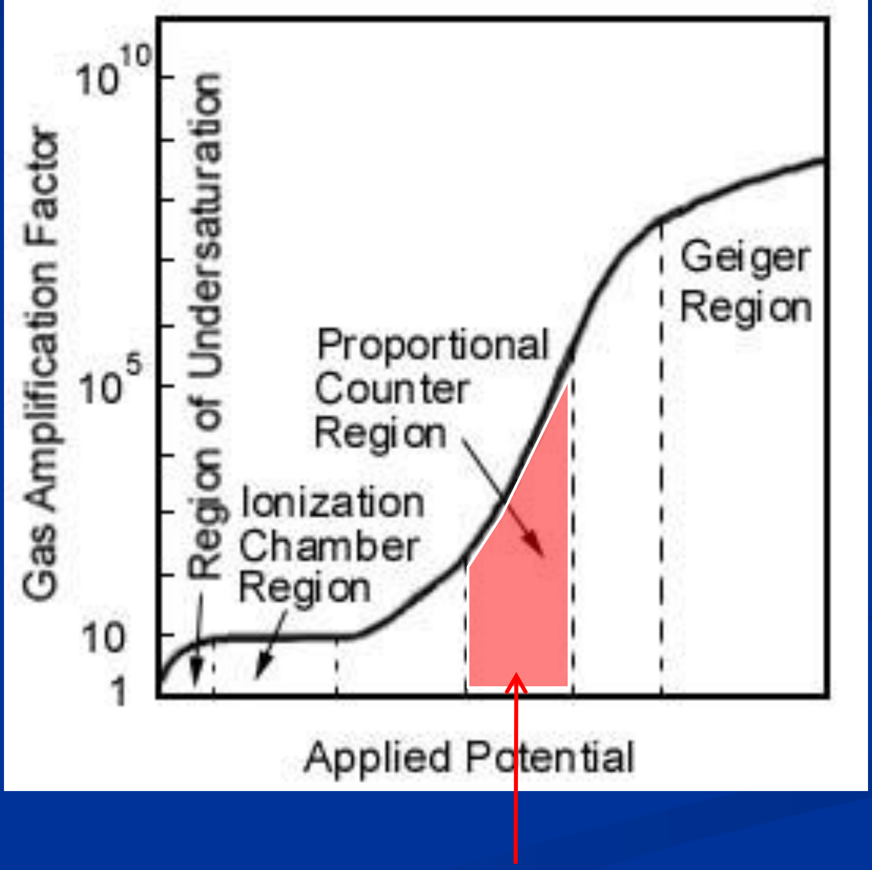
WDS detector: Proportional counter

Count rate depends on bias and gas used



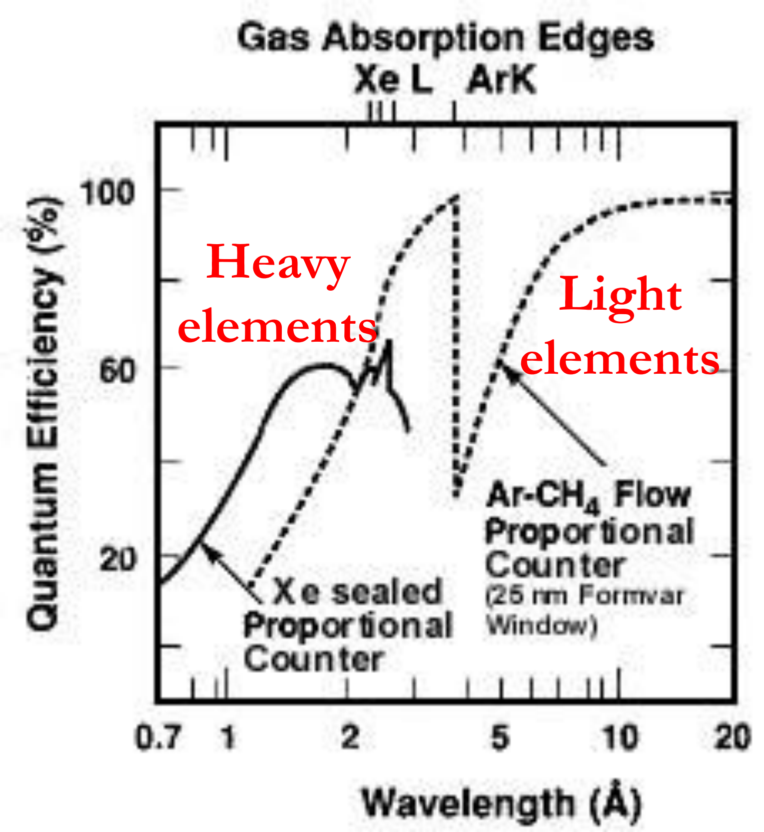
- *Tungsten collection wire set at 1-3 kV bias*
- *Flow counter: 90% Ar +10% CH_4 (P-10); poly-propylene window*
- *Sealed counter: Xe or Kr; Be window*

Bias in proportional counter



best count rate

Counting efficiency of gas



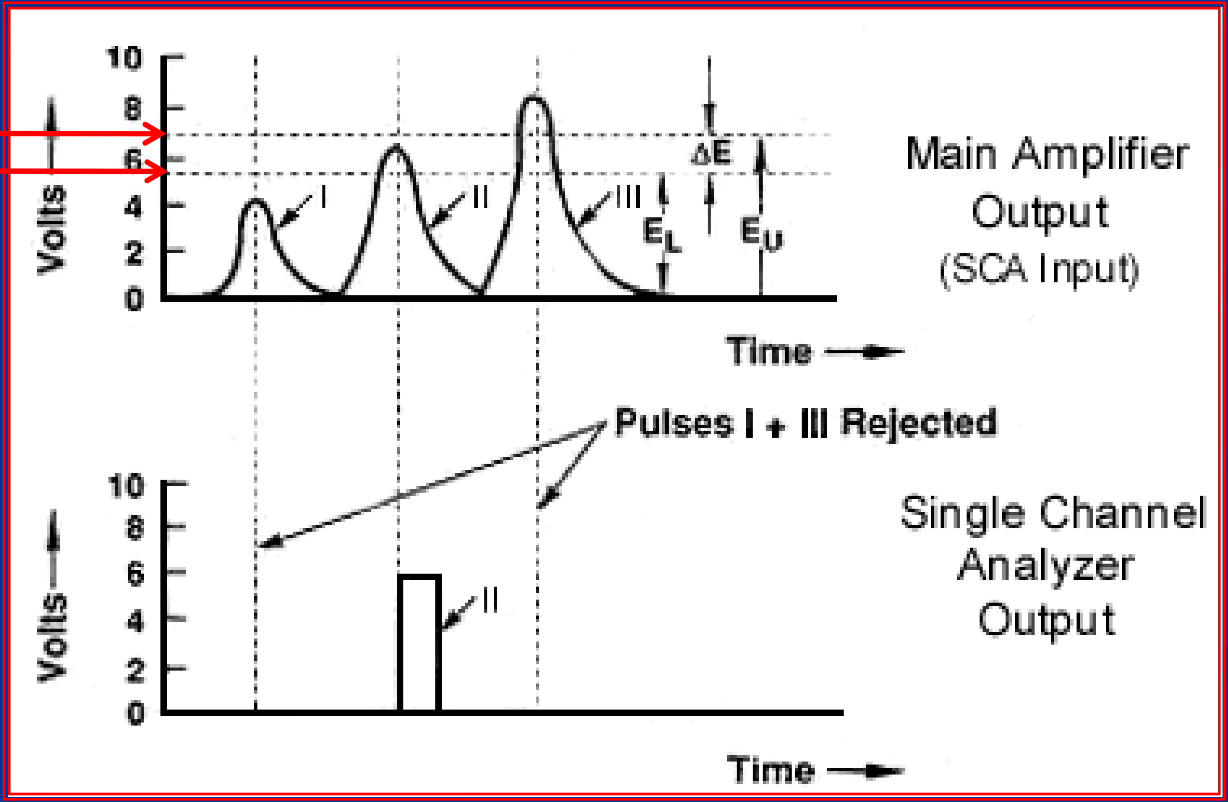
WDS signal processing

Single channel analyzer (SCA)
and
pulse height analysis (PHA)

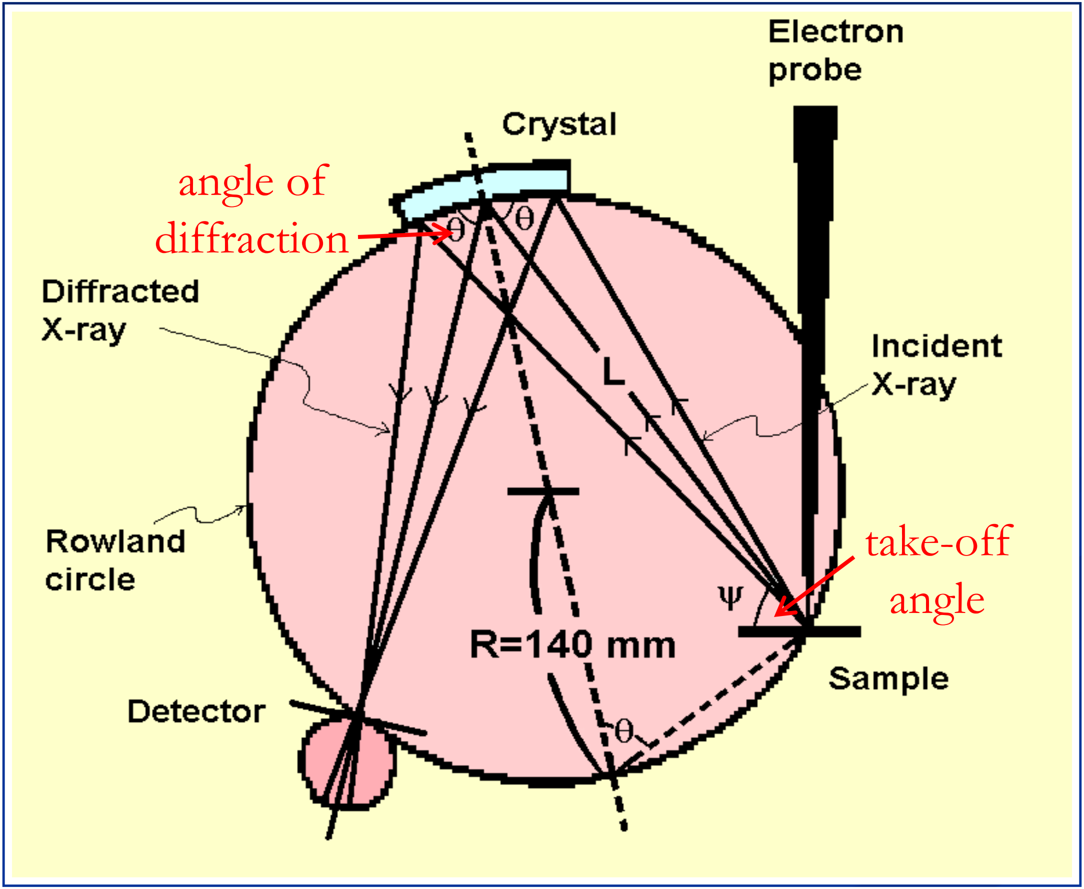
window

baseline

Only pulses in this voltage interval are counted

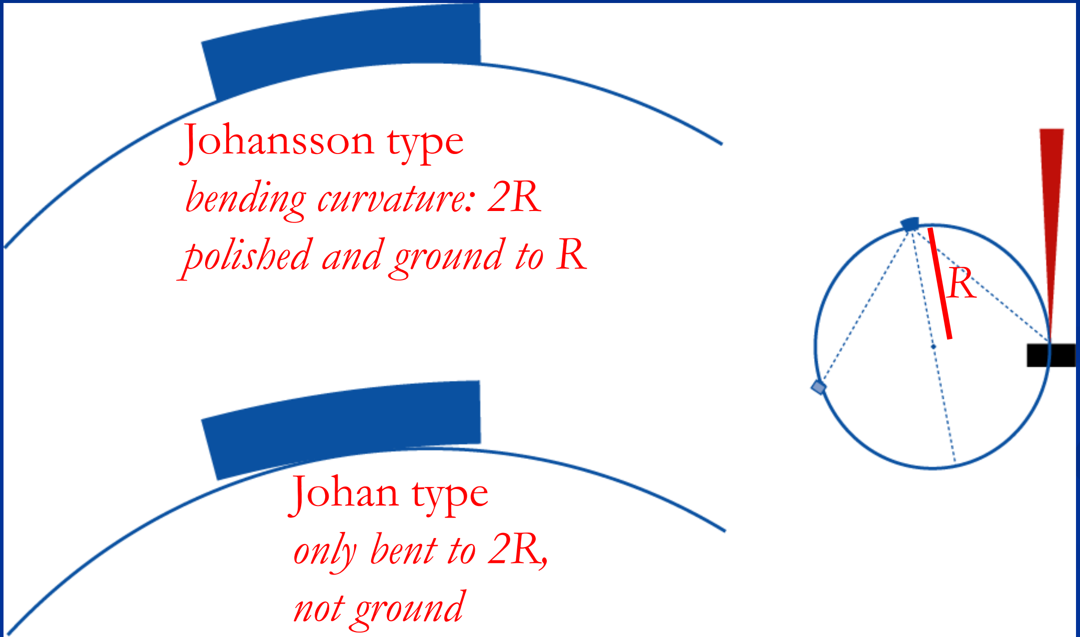


WDS Focusing geometry



$$L = n\lambda . R/d$$

Curved diffracting crystals



Johansson type

bending curvature: 2R

polished and ground to R

Johan type

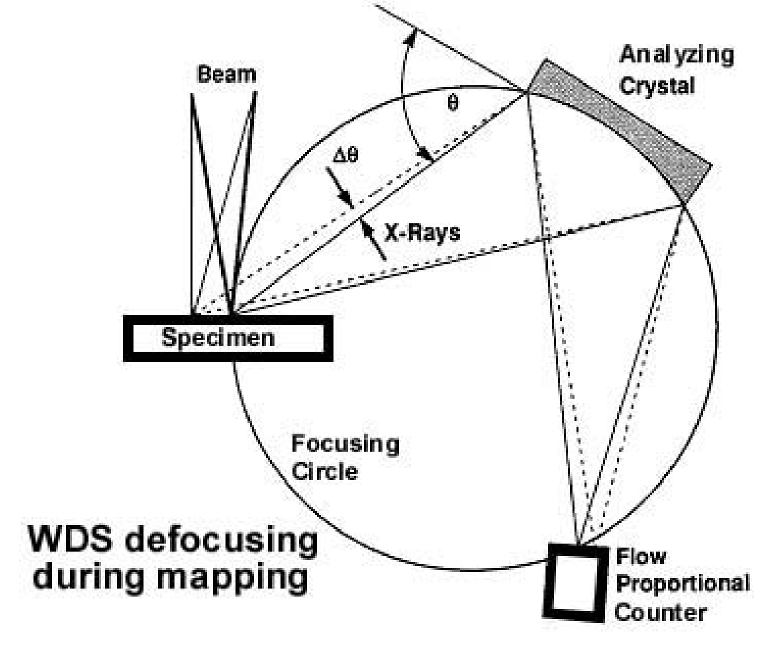
only bent to 2R,

not ground

FWHM of fully focusing Johansson-type crystal ~10 eV

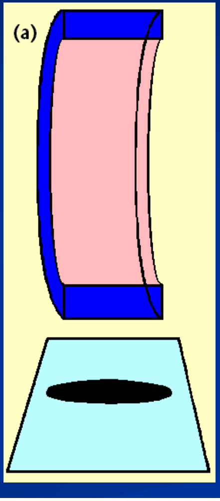
Some defocusing in Johan-type, but resolution is not compromised

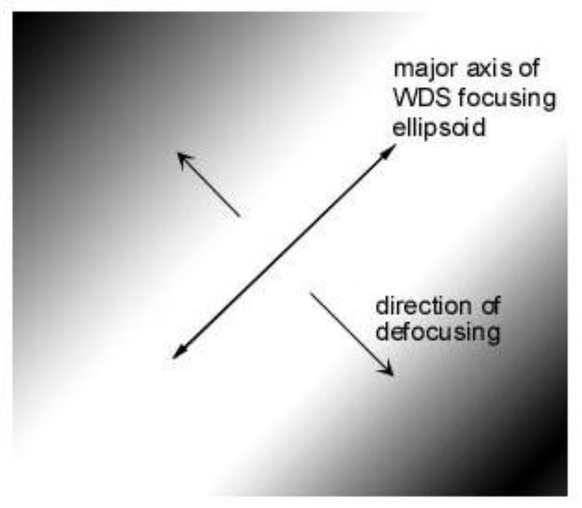
Defocusing in beam-rastered WDS X-ray maps



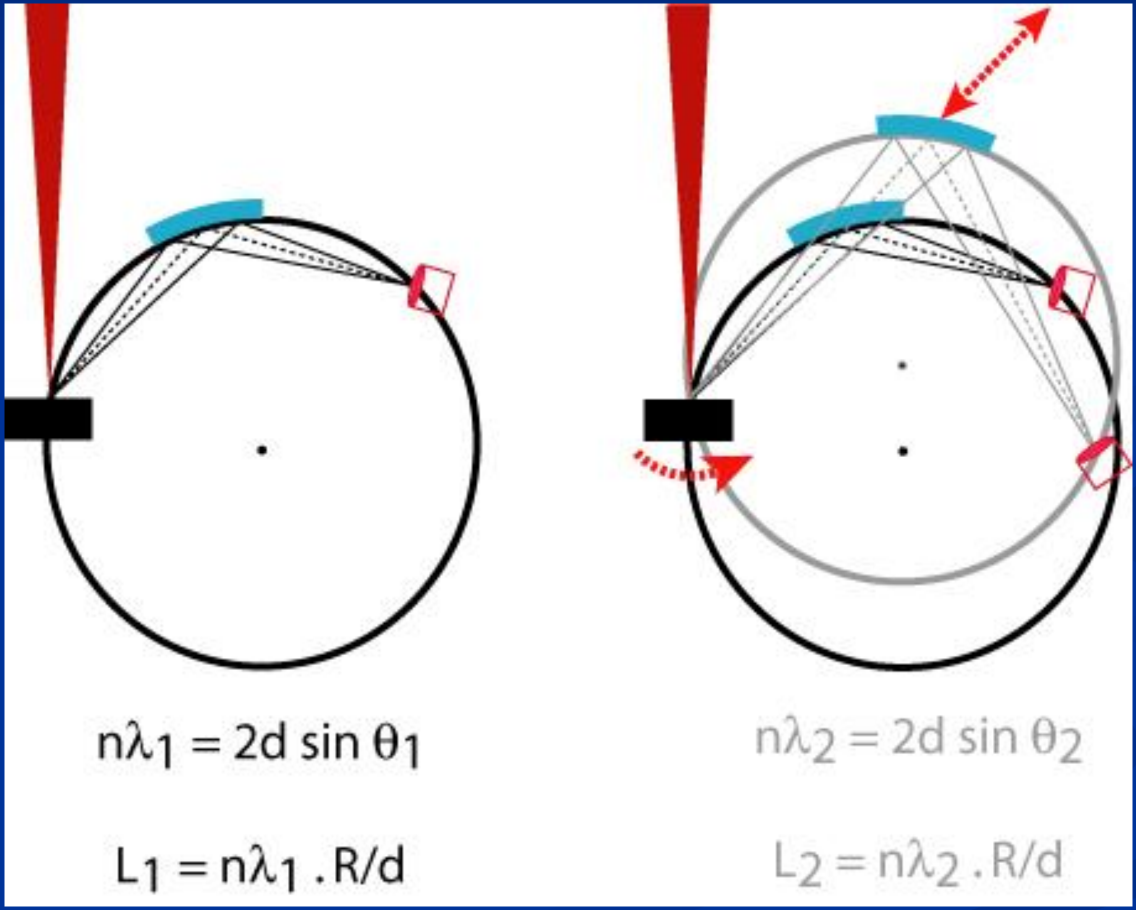
WDS defocusing during mapping

As the beam moves off the optic axis, the displacement in the specimen plane is equivalent to a change in the angle of incidence of the x-rays on the crystal by an angle Δθ.





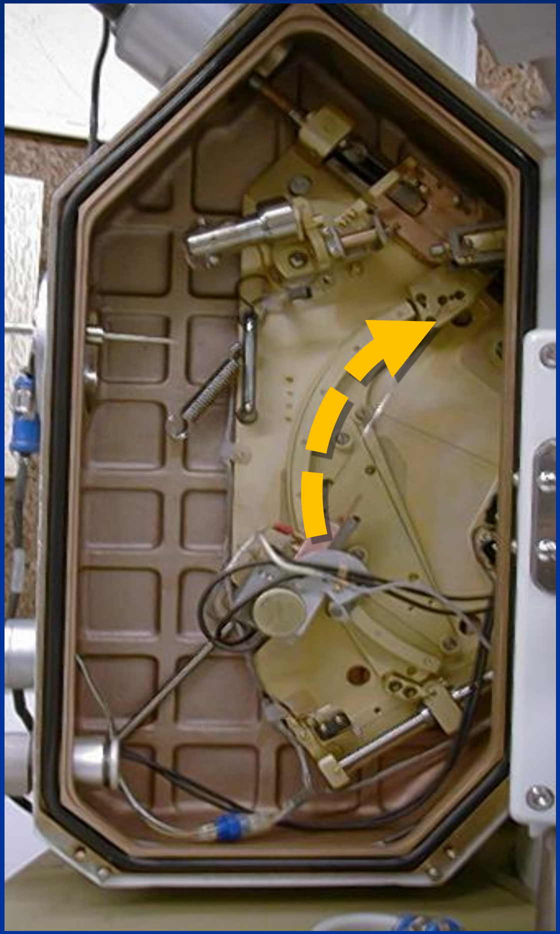
WDS: changing the angle of diffraction

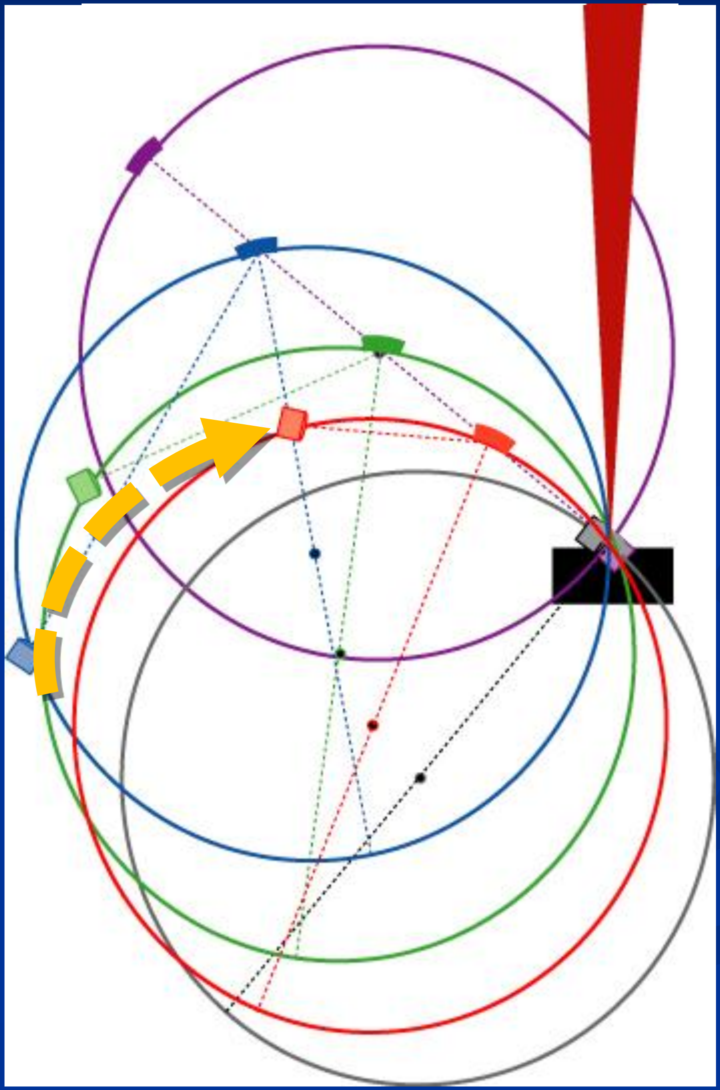


or, $n_2\lambda_1 = 2d \sin \theta_2$

$L_2 = n_2\lambda_1 . R/d$

Theoretical and actual limits of spectrometer movement





2R ≤ L ≤ 0

EPMA: Quantitative analysis procedure

- Sample preparation
- Qualitative analysis with EDS
- Standard intensity measurement (calibration)
- Measurement of X-ray intensities in the specimen
- Data reduction through matrix corrections

Sample preparation

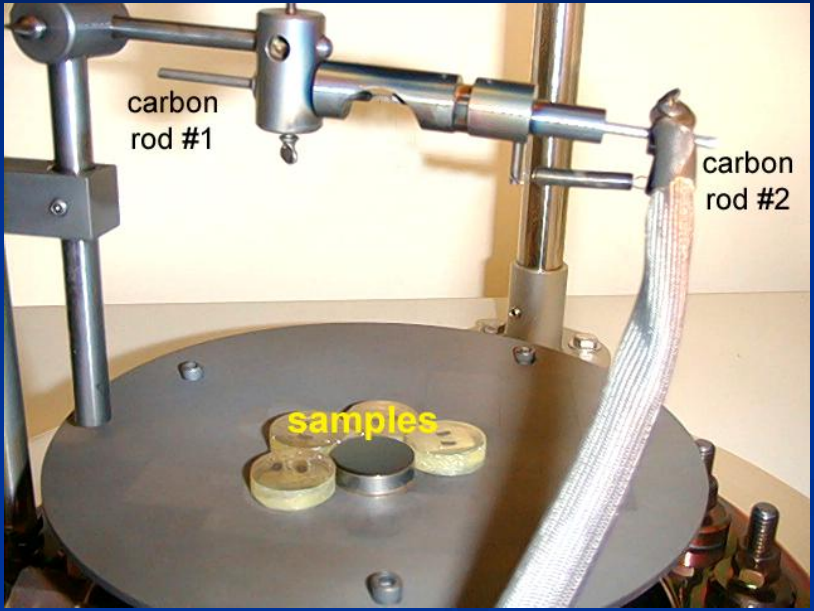
- Sample cut and mounted in epoxy
- Polished first with coarse SiC paper, then with alumina grit slurry (final size: ≤0.25 μm) [1]
- Washed with water in ultrasonic cleaner [2]
- Dried with blow duster and air
- Carbon coated [3]

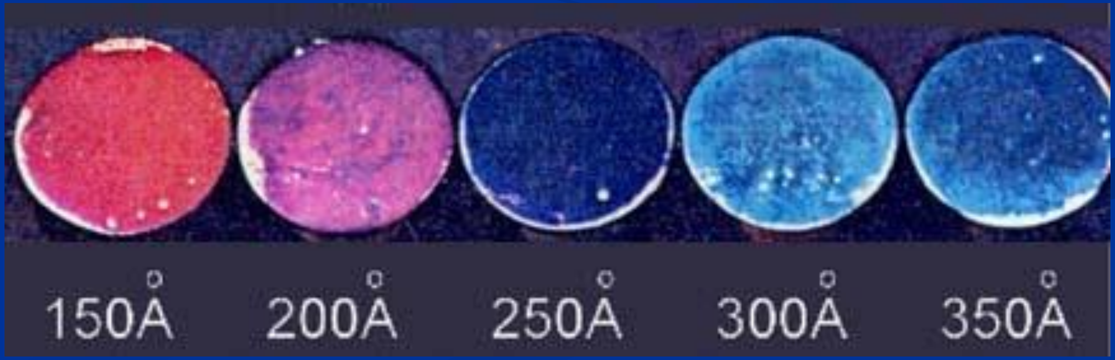
1: diamond paste or colloidal silica for some samples; dry polishing paper for water-soluble samples

2: ethanol may be used sparingly; cleaned with blow duster and cloth for samples that dissolve in water

3: for insulators; if standards are coated, however, all samples must be coated

Sample prep: carbon coating





12.119 Analytical Techniques for Studying Environmental and Geologic Samples
Spring 2011