XRD-5

X-ray diffractometer

Monochromators

Monochromatic radiation

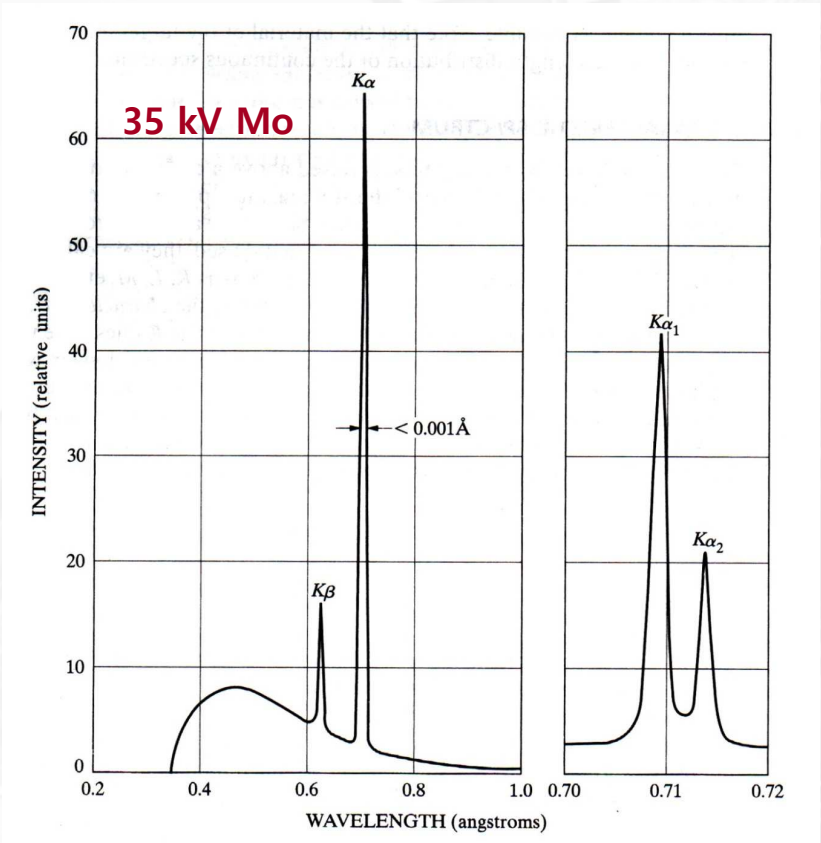
- Problems caused by polychromatic nature of diffracted beam & variability of angular dispersion
- XRD pattern from multiple wavelength, or that from unknown wavelength → difficulty in interpreting the pattern
- Why monochromatic beam is wanted? – we want to obtain experimental pattern from a single wavelength
- Monochromatization by reducing the intensity of white radiation & by eliminating undesirable characteristic wavelengths from X-ray spectrum
  - ✓ β filter
  - ✓ Diffracted beam monochromator
  - ✓ Primary beam monochromator
  - ✓ Pulse height selection using proportional counter
  - ✓ Use of solid state detector (high resolution energy resolving detector)

X-ray spectrum of molybdenum

2p → 1s

α1, α2 doublet
β1, β3 doublet

3p → 1s



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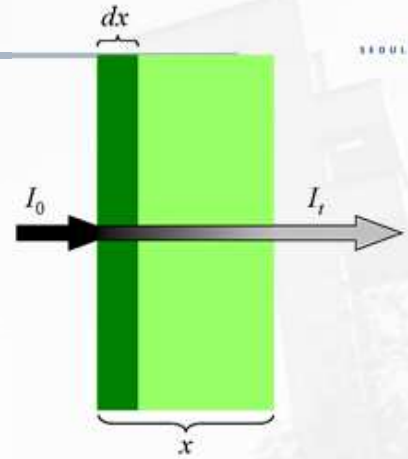
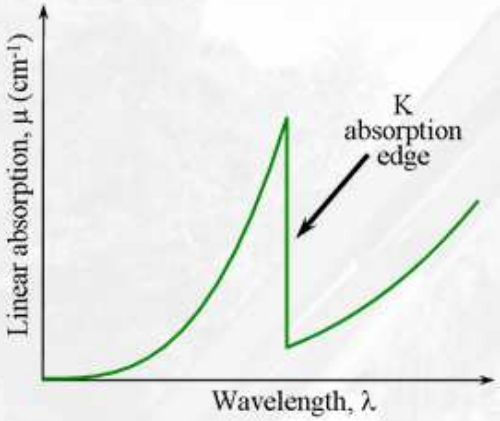
Absorption of X-rays

$$I_x = I_0 e^{-(\mu/\rho)\rho x}$$

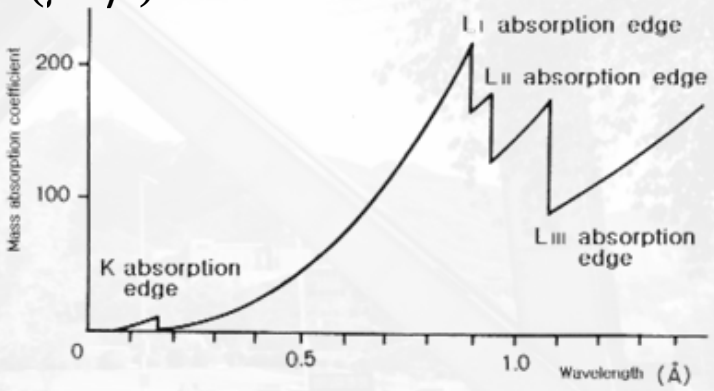
μ linear absorption coefficient

μ/ρ mass absorption coefficient

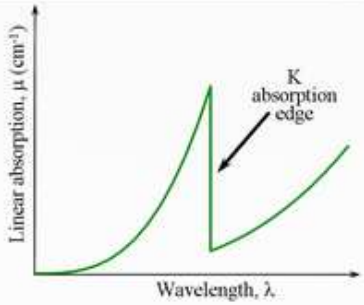


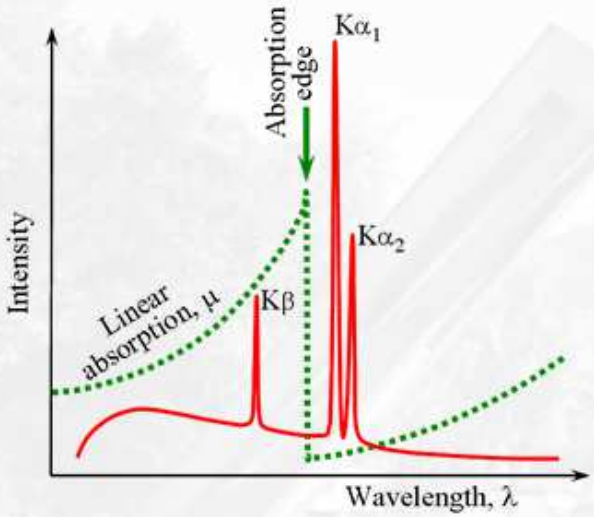


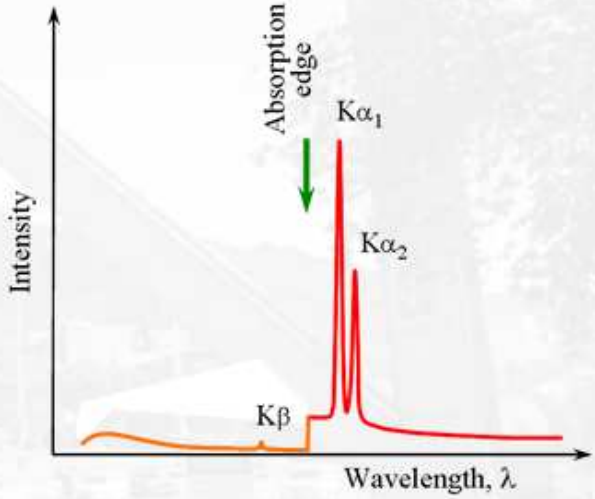
(μ/ρ)

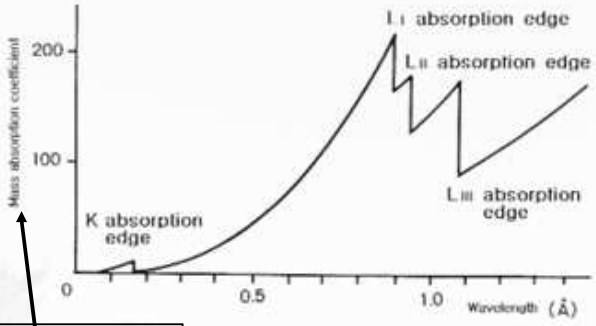


β-filter



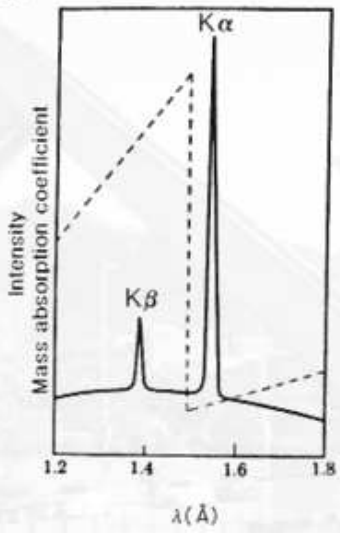


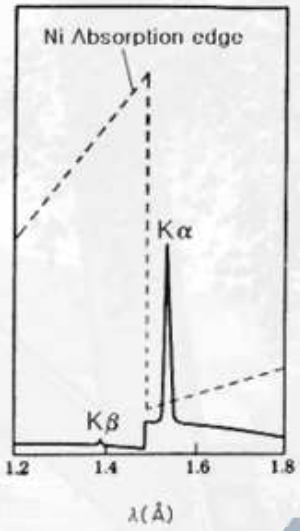




$$(\mu/\rho)$$

$$I_x = I_0 e^{-(\mu/\rho)\rho x}$$

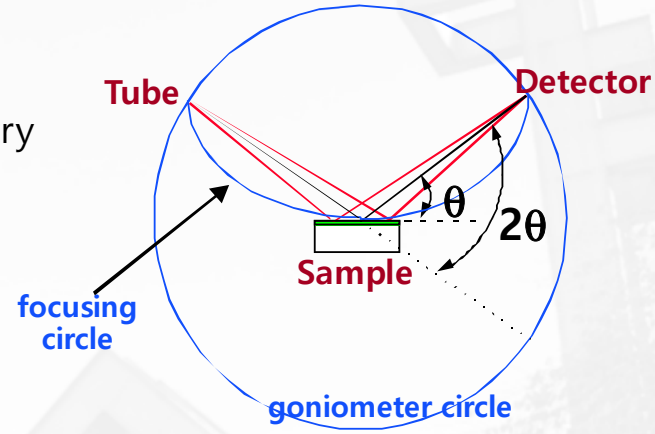


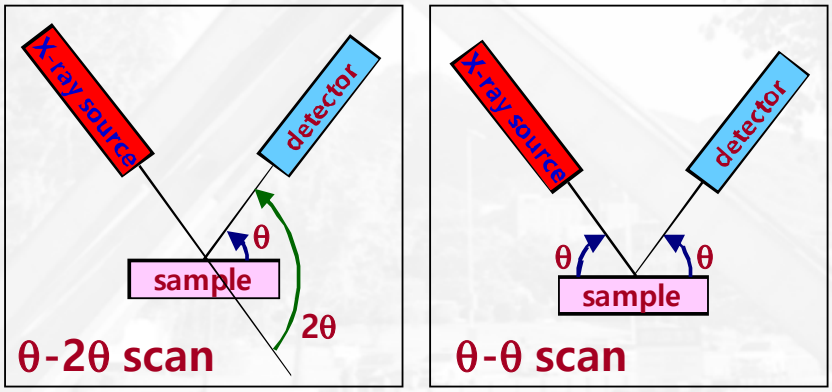


X-ray Diffractometer

X-ray Diffractometer

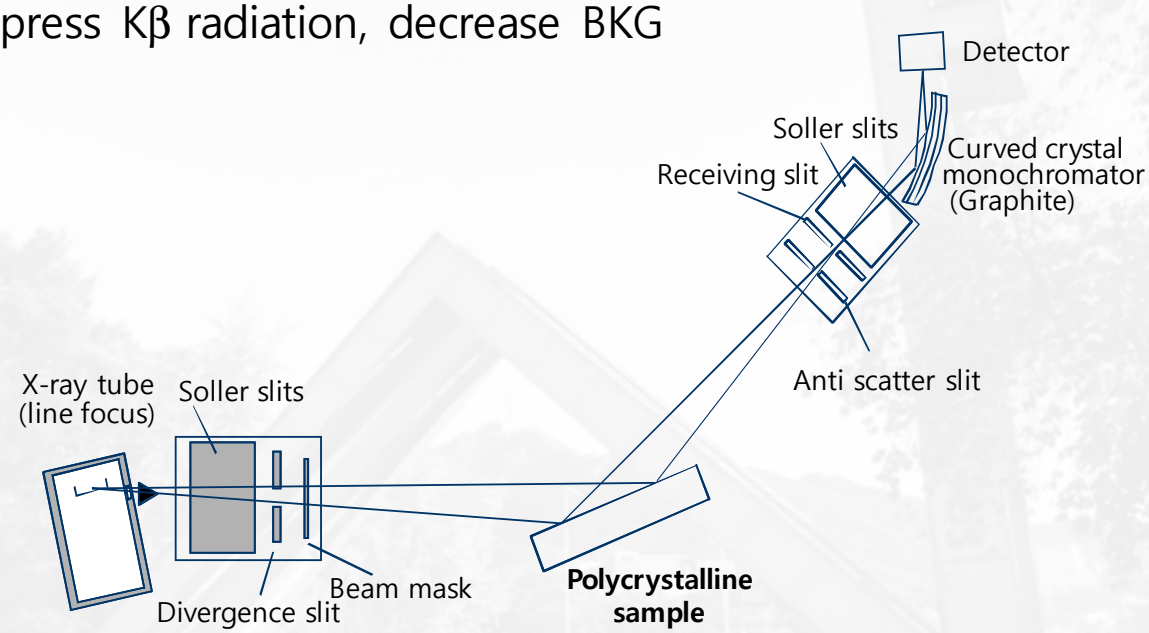
Bragg-Brentano Geometry
Parafocusing Geometry





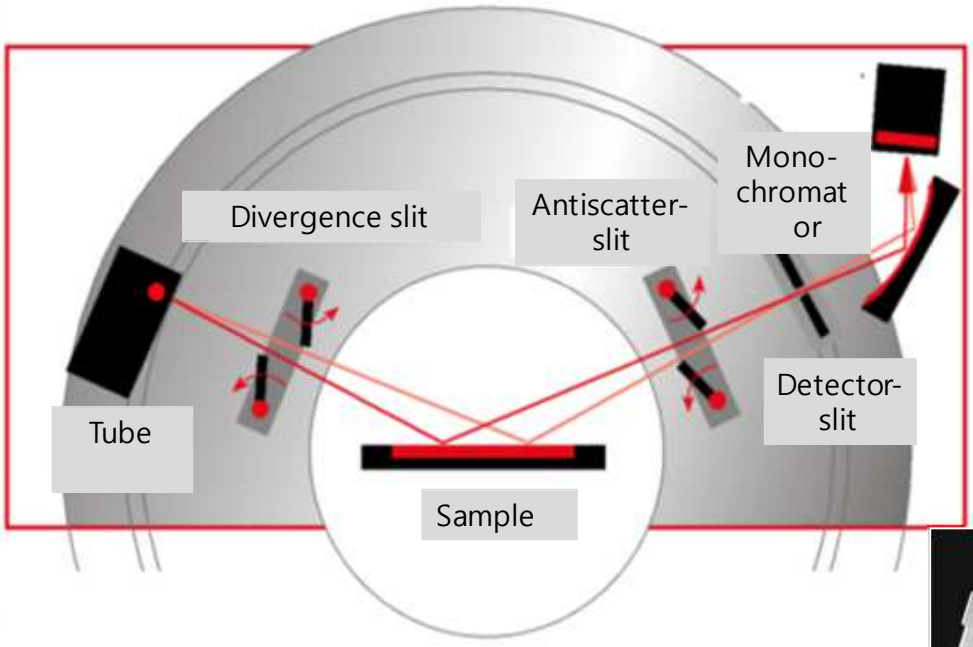
X-ray diffractometer; Bragg-Brentano

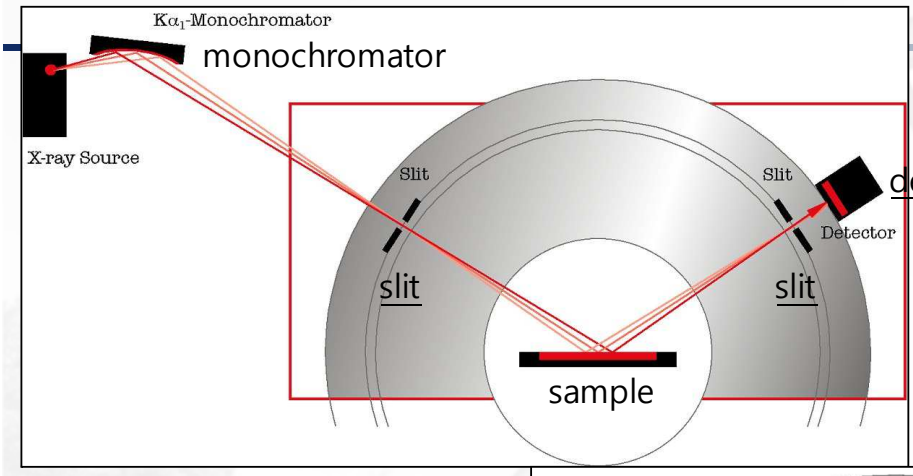
- monochromator or filter in diffracted beam - suppress Kβ radiation, decrease BKG

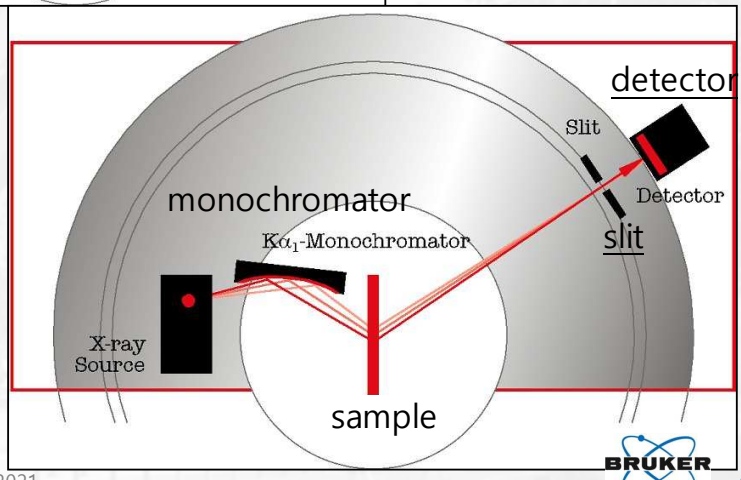


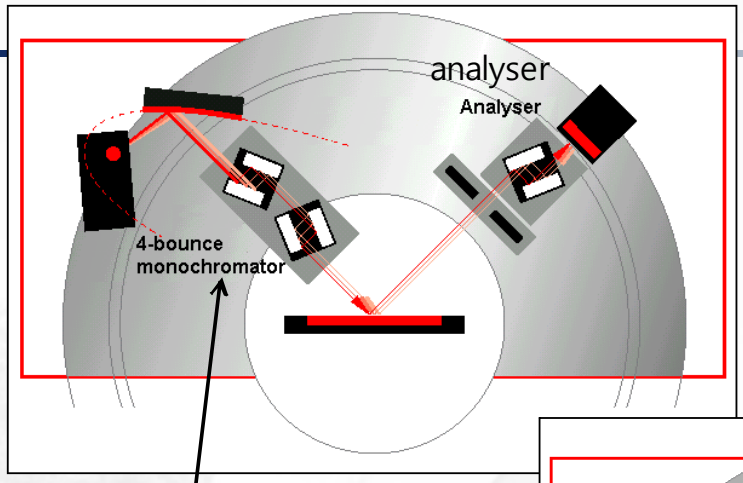
Phillips

The Bragg-Brentano Geometry

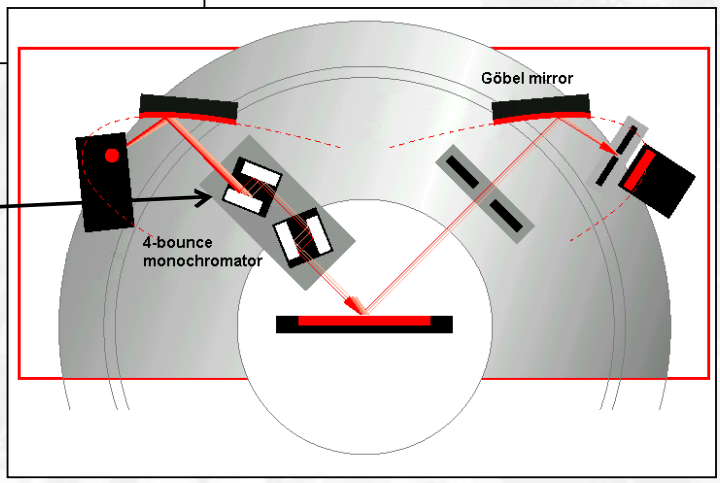






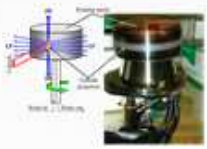


Göbel Mirror

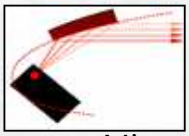


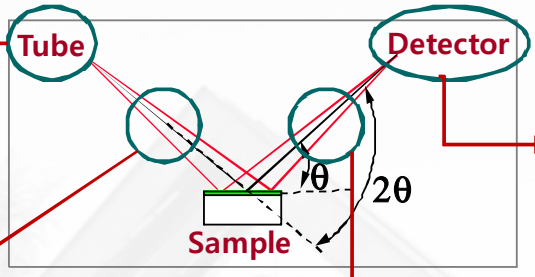
4-bounce
monochromator

X- ray Diffractometer



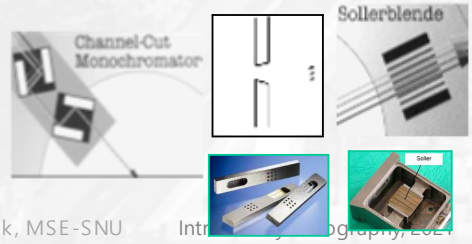
X선; Cu, Co, Cr, Mo,--- ???
Sealed tube, rotating anode ???





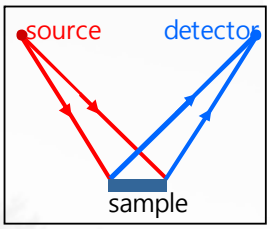
Point D, Line D, Area D ???
energy resolution ???
2 theta range ???

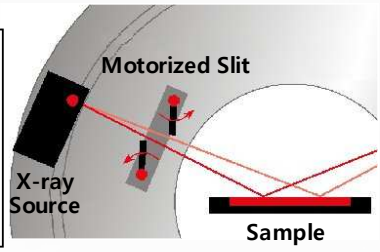
Mirror
Monochromator (graphite, Ge, Si)
Slit (primary slit, secondary slit)
Soller slit (primary, secondary SS)

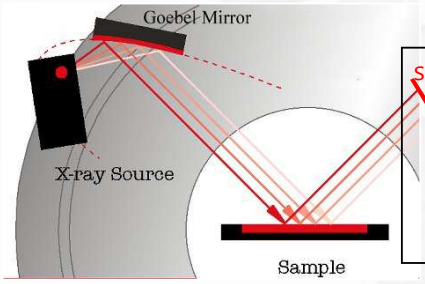


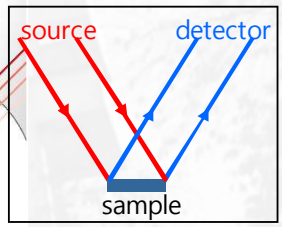
- Various combination is possible.
- The combination can (has to) be determined based on the purpose of the analysis.

Bragg-Brentano vs. Parallel Beam Geometry



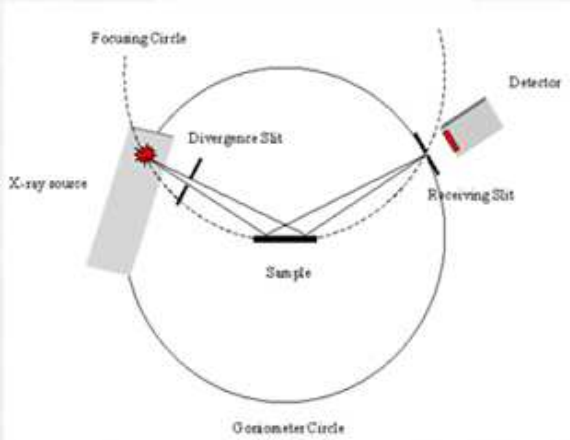


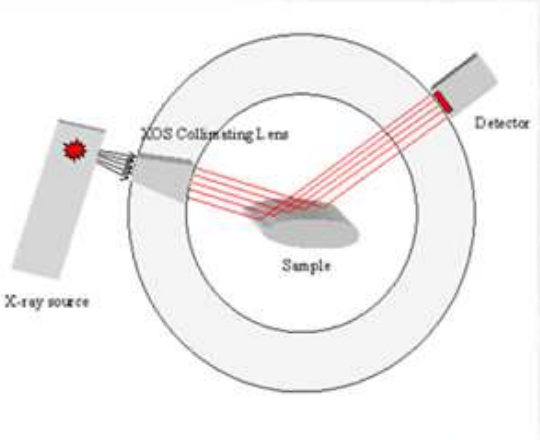




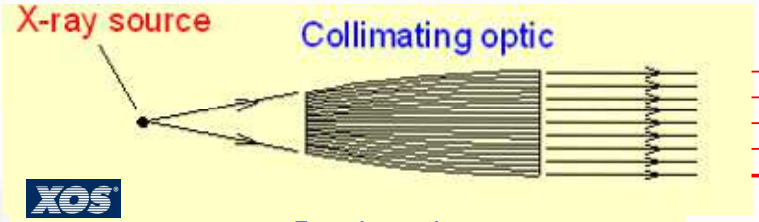
Bragg-Brentano Geometry
(para-focusing geometry)

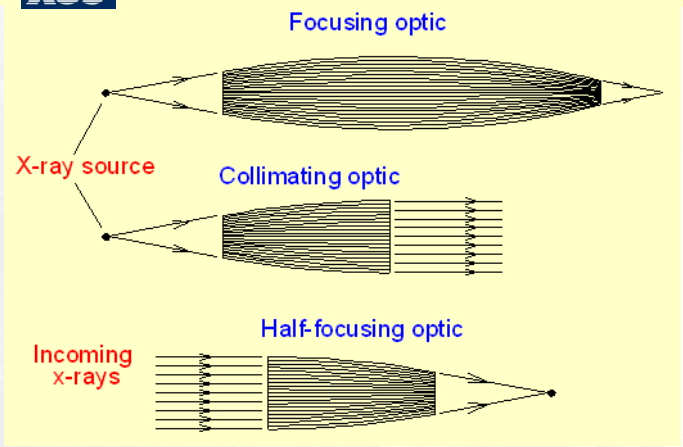
Parallel Beam Geometry
generated by Göbel Mirrors

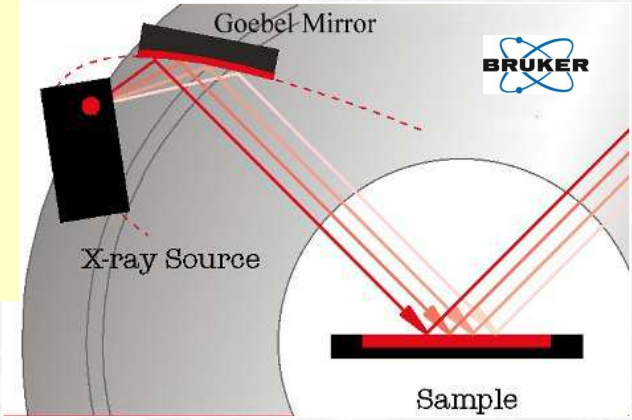




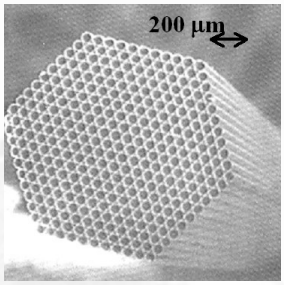
Parallel beam optics → no sample displacement error

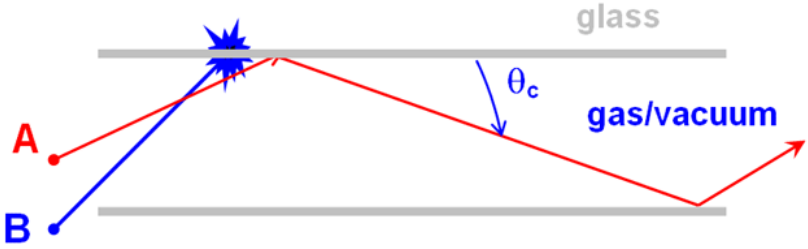






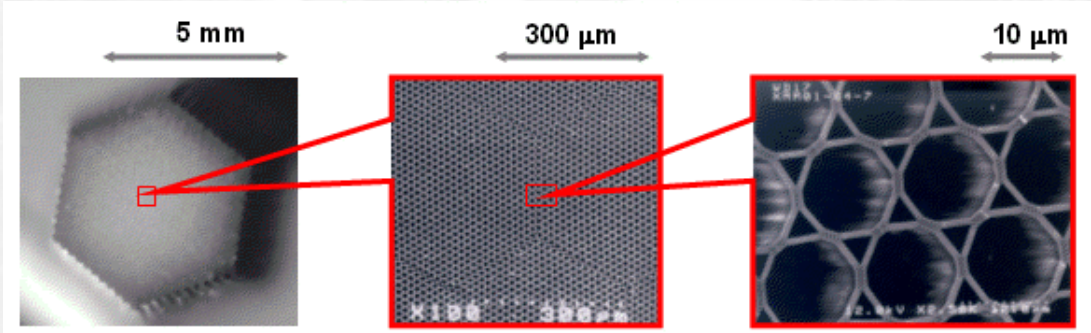
X-ray optical systems





Incident photon "A" angle ≤ critical angle (θ_c)
Incident photon "B" angle > critical angle (θ_c)

$$\theta_c\,(mrad) \cong \frac{30}{energy\,(keV)}$$



Parallel beam geometry can be used for

- analysis of samples with non-flat surfaces, e.g. corrosion on pipes
- samples you would prefer not to grind to a powder, e.g. jewelry, archaeology or forensic samples

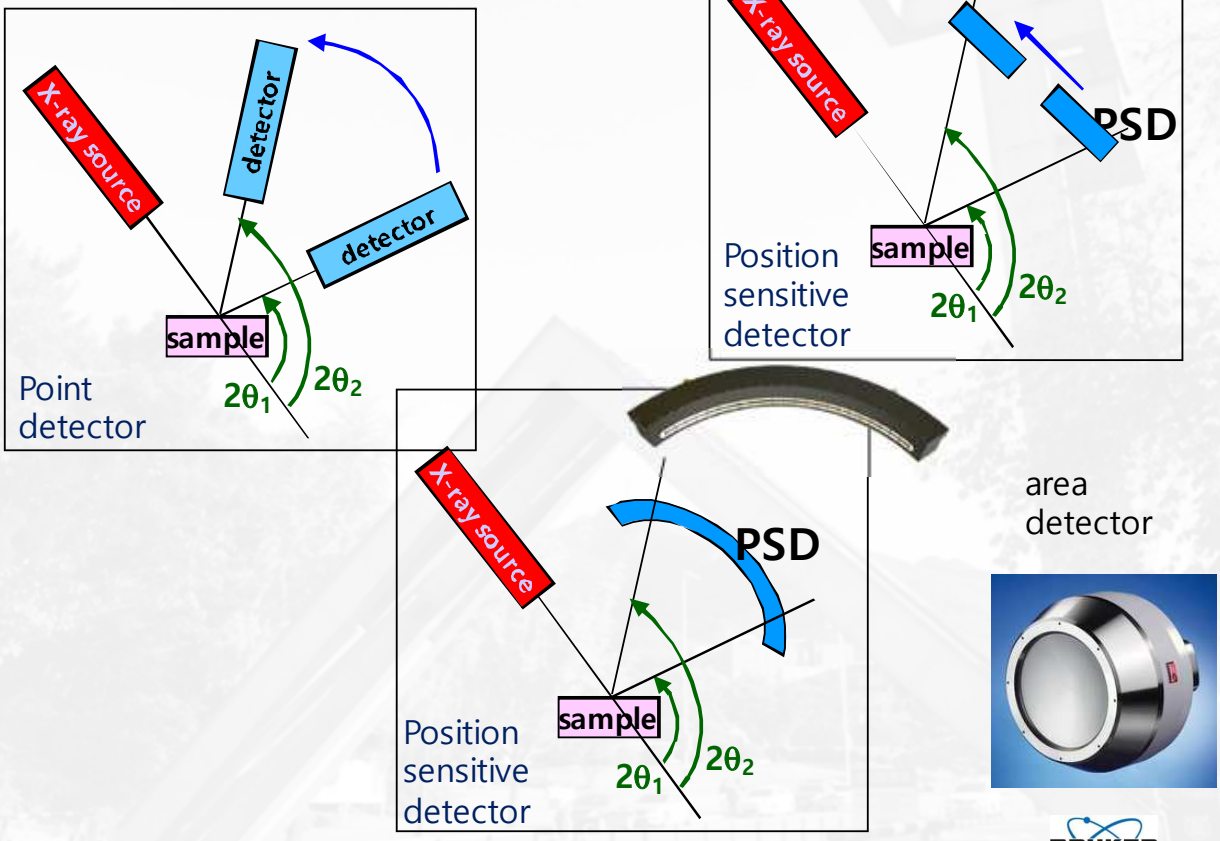
Sodalite Bracelet

Vesuvianite Pebble

- measure thermal expansion and contraction when using the heating/cooling stage
- grazing incidence diffraction (GID) of layers on substrates
- reflectometry for thin film thickness and roughness

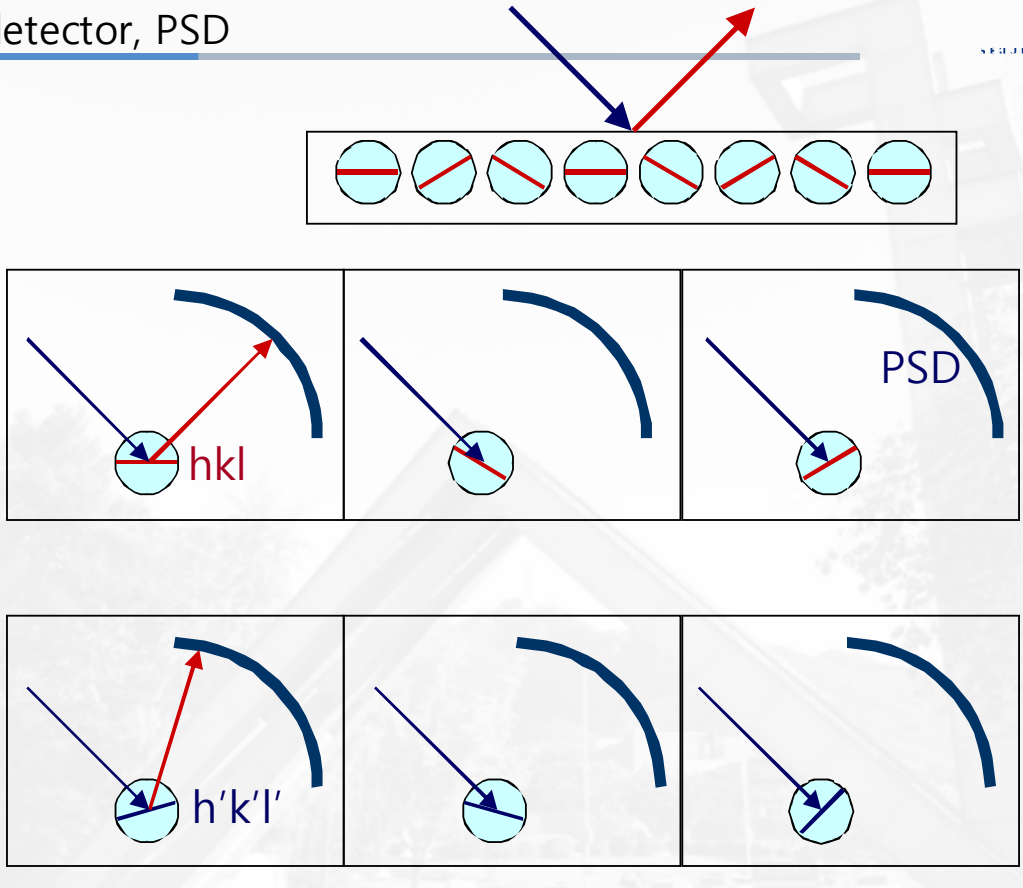
http://www.csec.ed.ac.uk/Instruments/D8_diffractometer/D8_parallel-beam.html

Point, Line, & Area detector

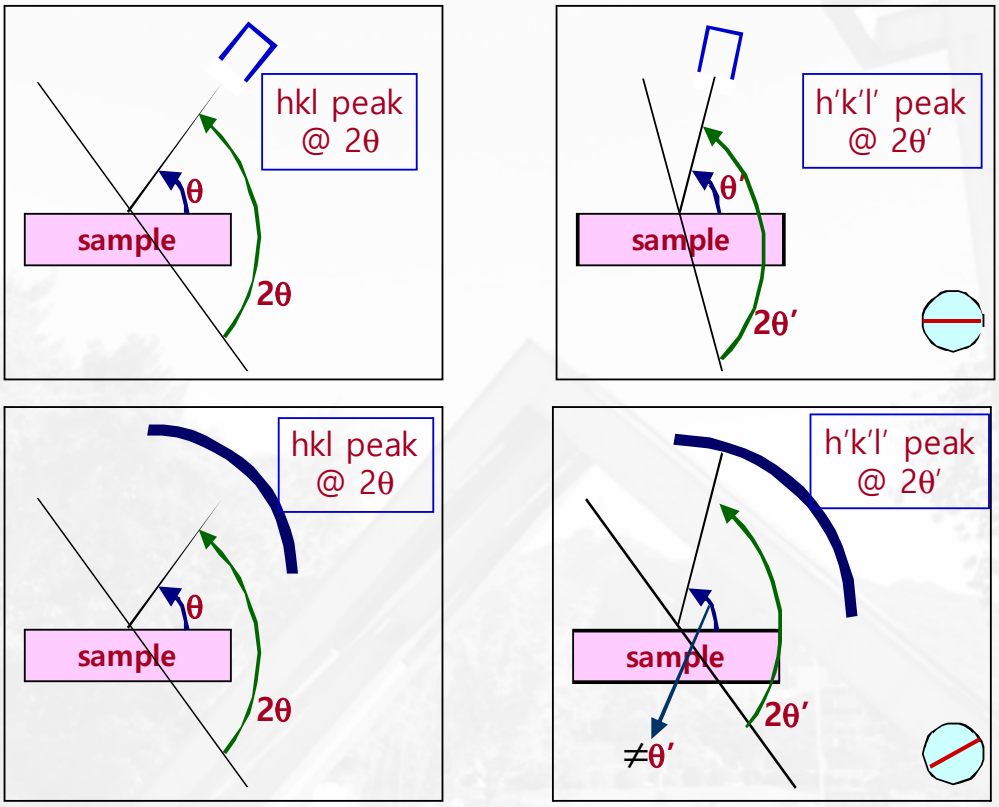


area detector

Line detector, PSD

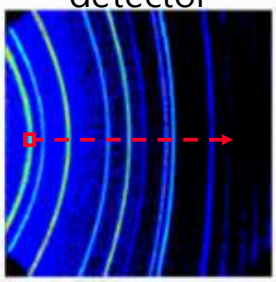


Point detector vs. Line detector



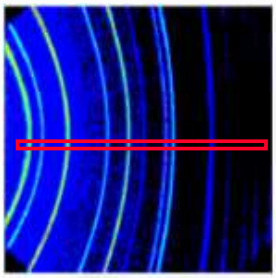
Point, Line, and Area detector

scintillation detector



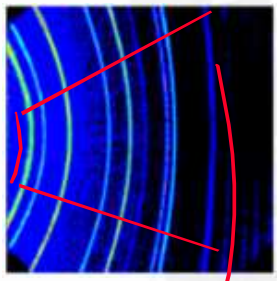
- ✓ small spot measured
- ✓ scan necessary
- ✓ long collection time

Line detector (PSD)

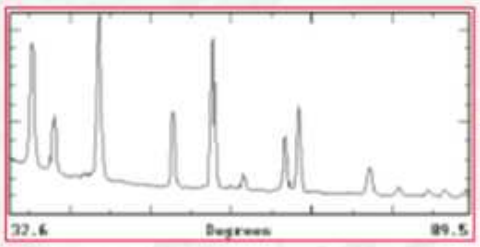


- ✓ large 2θ range measured simultaneously
- ✓ medium collection time

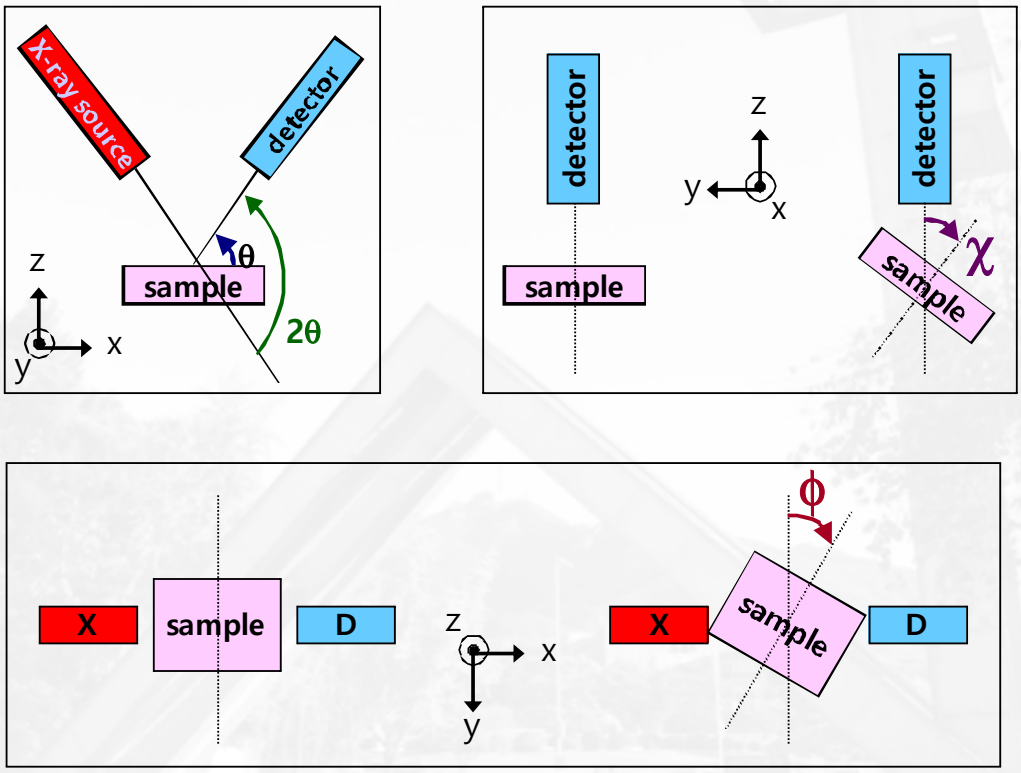
GADDS

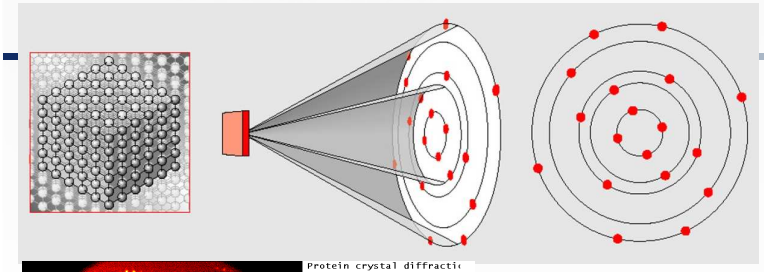


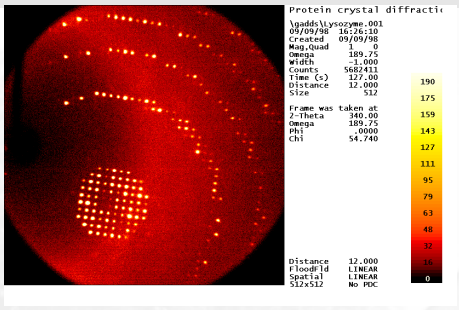
- ✓ large 2θ and chi range measured simultaneously
- ✓ measurement of oriented samples
- ✓ very short collection times
- ✓ intensity versus 2θ by integration of the data



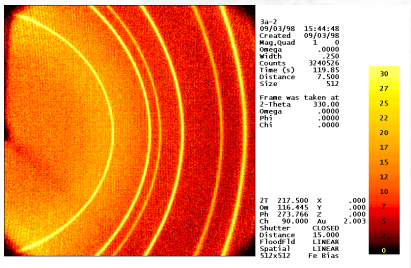
Angles - θ(w), 2θ, ϕ, χ



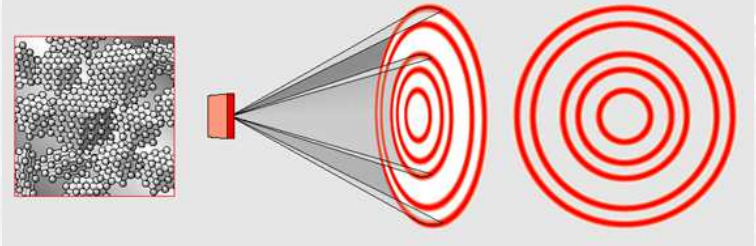




Single Crystal Diffraction

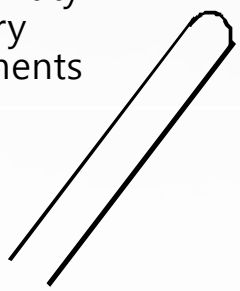


Powder Diffraction



Microdiffraction

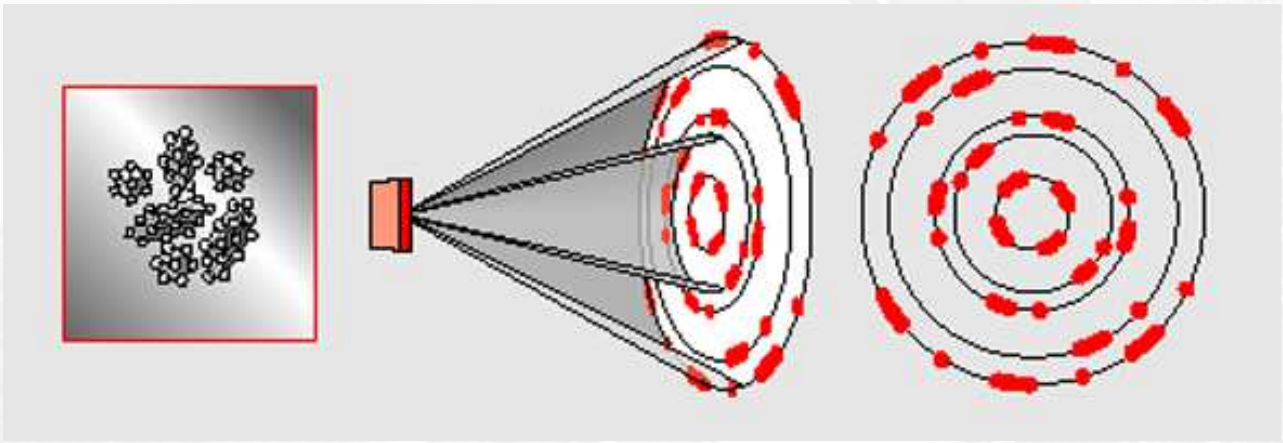
"micro-quantity"
capillary
measurements

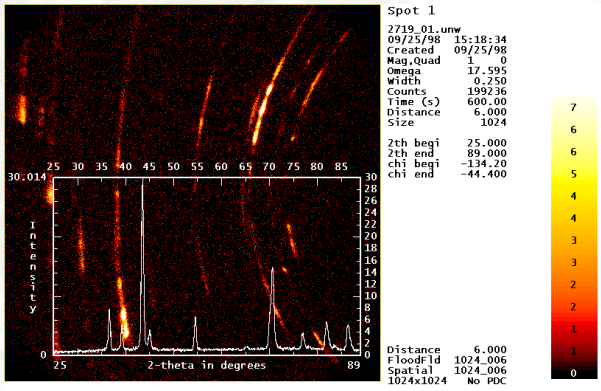


"micro-area"
X-ray microprobe

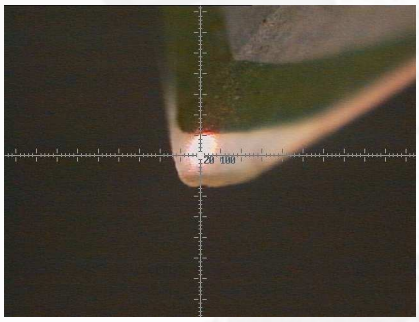
- Inspection of a Gold Pad
  - ✓ qualitative phase analysis
  - ✓ micro-texture
  - ✓ quick point-and-shoot operation

Microdiffraction

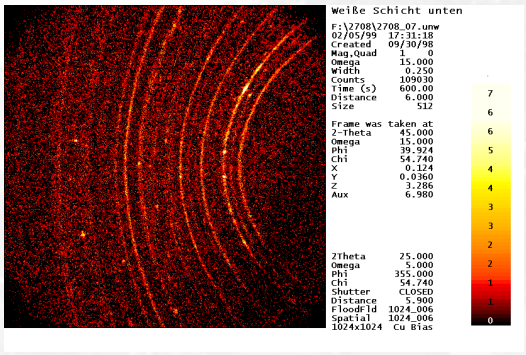




Forensic Application > Fatal Bicycle Accident Collection of Evidence



traces of car paint
found on the bicycle



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BRUKER