



CHEMISTRY 5570

Advanced Analytical Chemistry

X-ray Diffraction

Lecture 5

Dr. Teresa D. Golden

University of North Texas

Department of Chemistry

Advance X-Ray Diffraction

Class Website:

https://sites.chemistry.unt.edu/~tgolden/courses/course_downloadsFall24.xhtml

Readings:

Given at the end of each powerpoint lecture. The books are on reserve at the Willis library under CHEM 5390 (X-ray Diffraction).

Homework Assignments:

Given at the end of each powerpoint lecture. I do not accept assignments by email – all assignments must be turned in during class.

Exams:

There will be an exam in class on Tuesday, December 10th, 8:00 - 10:00 a.m.



Important for Calculations

$$n\lambda = 2d \sin \theta$$

where n is an integer

λ is the wavelength of the x-rays

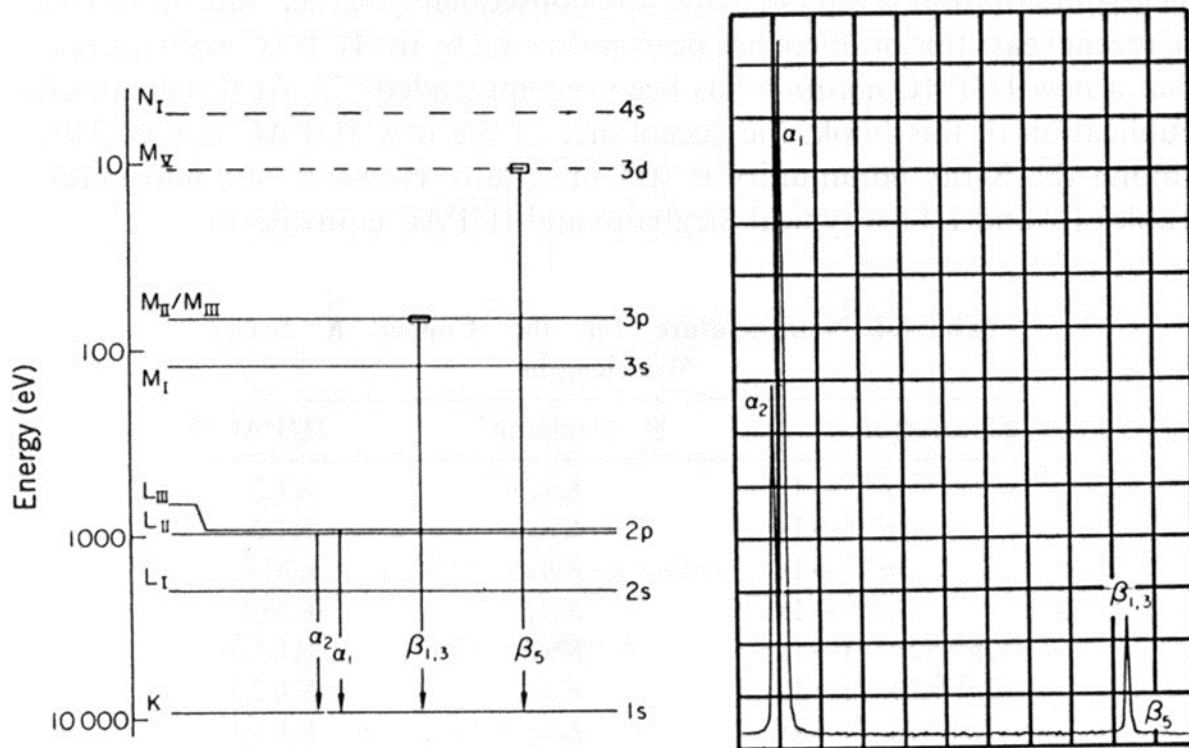
d is the interplanar spacing in the specimen

θ is the diffraction angle

Bragg Equation

Properties of X-rays

The Copper K Spectrum



The copper $K\alpha$ spectrum.

- The diagram at left shows the 5 possible Cu K transitions
- L to K “jumps:
 - $K\alpha_1$ (8.045 keV, 1.5406Å)
 - $K\alpha_2$ (8.025 keV, 1.5444Å)
- M to K
 - $K\beta_1$ $K\beta_3$ (8.903 keV, 1.3922Å)
 - $K\beta_5$

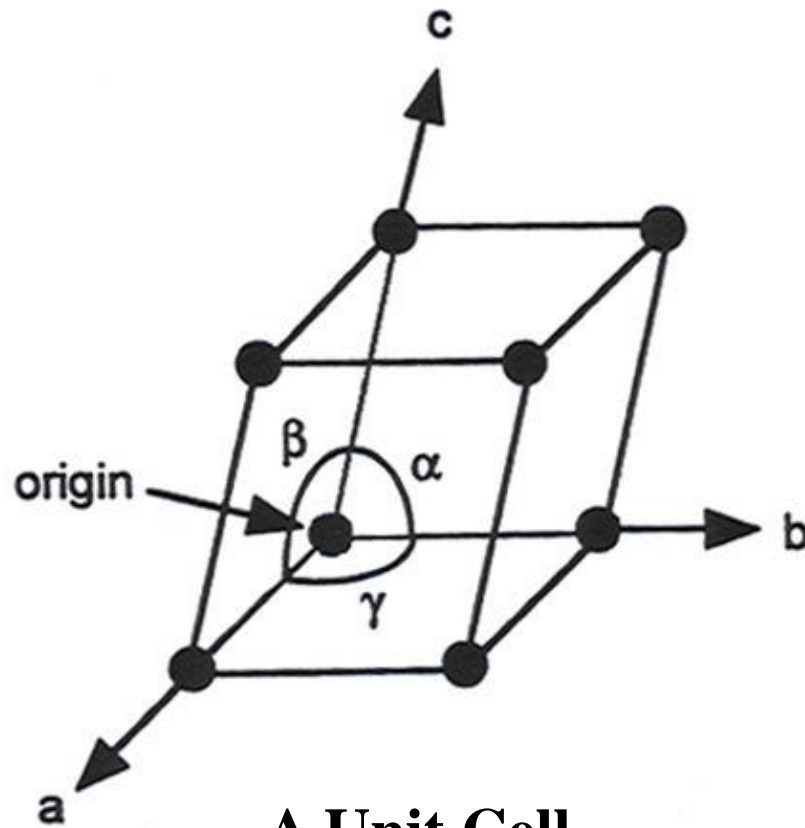
Crystallography

The size and shape of the unit cell can be described by three vectors, a , b , and c (called the crystallographic axes of the cell).

The unit cell can also be described in terms of lengths (a , b , c) and the angles between them (α , β , γ).

The lengths and angles are the lattice constants or lattice parameters of the unit cell.

Notice that the entire point lattice can be built by translating the unit cell.



A Unit Cell.

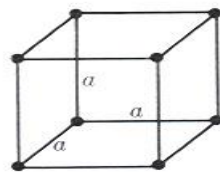
| Axis | a | b | c |
|---------------------|----------|----------|----------|
| Lattice Parameters: | | | |
| Lengths | a | b | c |
| Inter-axial angle | α | β | γ |

(The symbol \neq means that equality is not required by symmetry. Accidental equality may occur, as shown by an example in Sec. 2-4.)

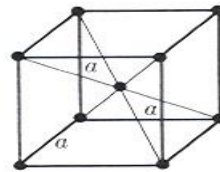
| System | Axial lengths and angles | Bravais lattice | Lattice symbol |
|---------------|---|---|------------------|
| Cubic | Three equal axes at right angles $a = b = c, \alpha = \beta = \gamma = 90^\circ$ | Simple Body-centered Face-centered | P I F |
| Tetragonal | Three axes at right angles, two equal $a = b \neq c, \alpha = \beta = \gamma = 90^\circ$ | Simple Body-centered | P I |
| Orthorhombic | Three unequal axes at right angles $a \neq b \neq c, \alpha = \beta = \gamma = 90^\circ$ | Simple Body-centered Base-centered Face-centered | P I C F |
| Rhombohedral* | Three equal axes, equally inclined $a = b = c, \alpha = \beta = \gamma \neq 90^\circ$ | Simple | R |
| Hexagonal | Two equal coplanar axes at 120° , third axis at right angles $a = b \neq c, \alpha = \beta = 90^\circ, \gamma = 120^\circ$ | Simple | P |
| Monoclinic | Three unequal axes, one pair not at right angles $a \neq b \neq c, \alpha = \gamma = 90^\circ \neq \beta$ | Simple Base-centered | P C |
| Triclinic | Three unequal axes, unequally inclined and none at right angles $a \neq b \neq c, \alpha \neq \beta \neq \gamma \neq 90^\circ$ | Simple | P |

* Also called trigonal.

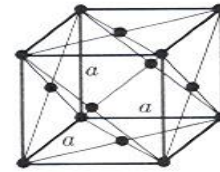
7 Crystal systems and 14 Bravais lattices.



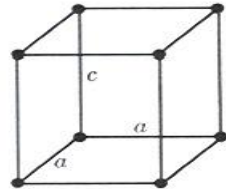
SIMPLE CUBIC (P)



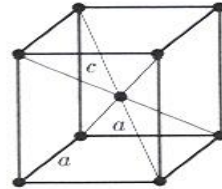
BODY-CENTERED CUBIC (I)



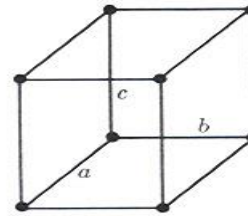
FACE-CENTERED CUBIC (F)



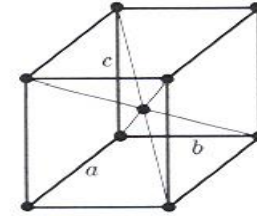
SIMPLE TETRAGONAL (P)



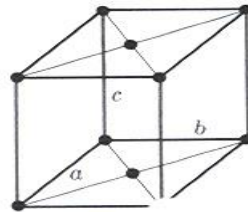
BODY-CENTERED TETRAGONAL (I)



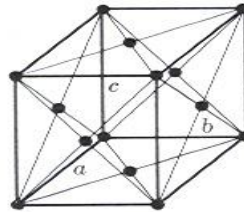
SIMPLE ORTHORHOMBIC (P)



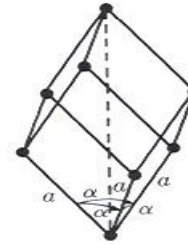
BODY-CENTERED ORTHORHOMBIC (I)



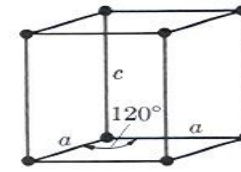
BASE-CENTERED ORTHORHOMBIC (C)



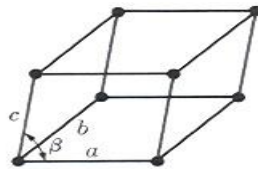
FACE-CENTERED ORTHORHOMBIC (F)



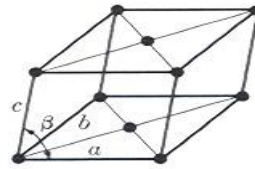
RHOMBOHEDRAL (R)



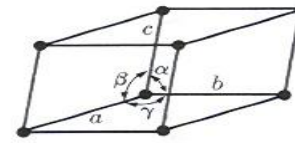
HEXAGONAL (P)



SIMPLE MONOCLINIC (P)



BASE-CENTERED MONOCLINIC (C)



TRICLINIC (P)

The fourteen Bravais lattices.

Crystallography

Geometry and the structure of crystals

Vectors and Planes

Miller Indices

A notation used to describe various planes within a crystal lattice.

Steps to determine Miller Indices

- 1) Identify the points at which the plane intersects the a, b, c axes. Intercept is measured in terms of fractions or multiples of the lattice parameter.
- 2) Take reciprocals of the intercepts. (Get rid of infinity)
- 3) Multiply to get a whole number (Clear the fractions)
- 4) Enclose numbers in (). Represent negative numbers with a bar (bar one).

Crystallography

Geometry and the structure of crystals

Vectors and Planes

In the cubic system there are six faces equivalent to $(1\ 0\ 0)$.

This set is related and denoted by $\{1\ 0\ 0\}$ - this set is called a family of planes.

$\{ \quad \}$ - denotes a family of planes

(\quad) - denotes an individual plane

The six planes in the $\{1\ 0\ 0\}$ family are:

$(1\ 0\ 0)$ $(0\ 1\ 0)$ $(0\ 0\ 1)$ $(\bar{1}\ 0\ 0)$ $(0\ \bar{1}\ 0)$ $(0\ 0\ \bar{1})$

Crystallography

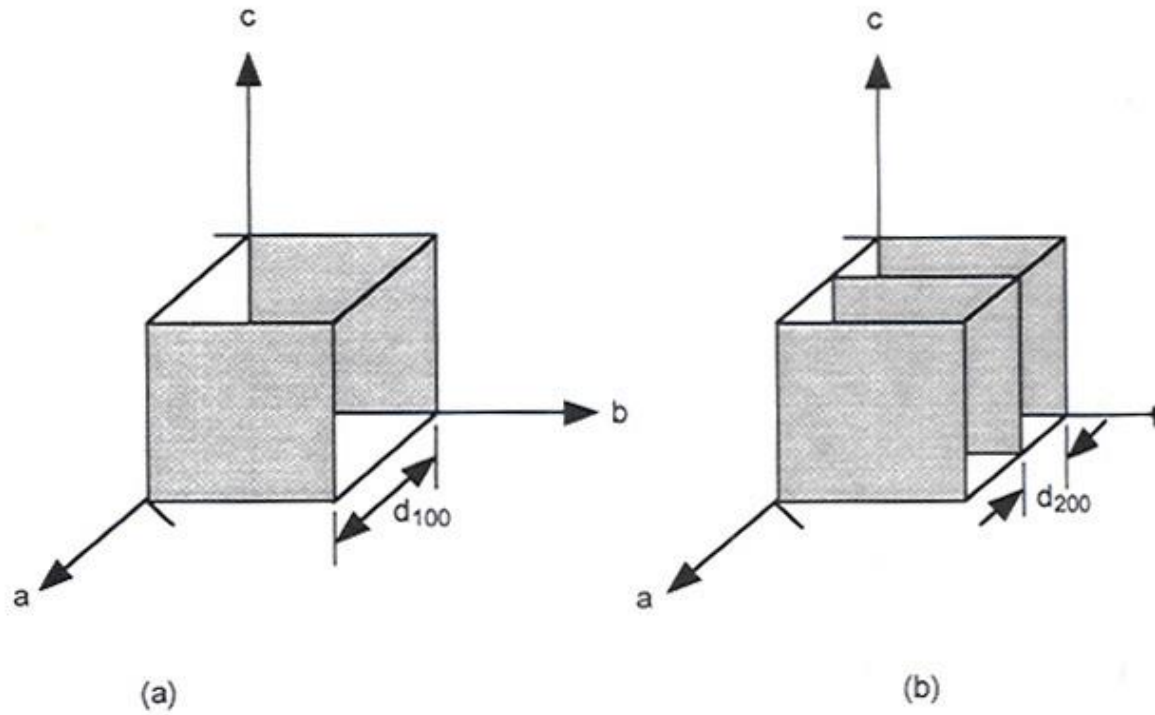
Geometry and the structure of crystals

Interplanar Spacings

The distance between an equivalent set of planes is defined as d_{hkl} - the interplanar spacing.

The interplanar spacing, d_{hkl} , measured at right angles to the planes, is a function both of the plane indices (hkl) and the lattice constants (a,b,c, α , β , γ).

The distance can be directly determined by x-ray diffraction.



The d_{hkl} interplanar spacing.

For a cubic system:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

The d_{hkl} interplanar spacing.

Cubic:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

Tetragonal:

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$$

Hexagonal:

$$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$$

Rhombohedral:

$$\frac{1}{d^2} = \frac{(h^2 + k^2 + l^2)\sin^2 \alpha + 2(hk + kl + hl)\cos^2 \alpha - \cos \alpha}{a^2(1 - 3\cos^2 \alpha + 2\cos^3 \alpha)}$$

Orthorhombic:

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$

Monoclinic:

$$\frac{1}{d^2} = \frac{1}{\sin^2 \beta} \left(\frac{h^2}{a^2} + \frac{k^2 \sin^2 \beta}{b^2} + \frac{l^2}{c^2} - \frac{2hl \cos \beta}{ac} \right)$$

Triclinic:

$$\frac{1}{d^2} = \frac{1}{V^2} (S_{11}h^2 + S_{22}k^2 + S_{33}l^2 + 2S_{12}hk + 2S_{23}kl + 2S_{13}hl)$$

V = volume of unit cell

$$S_{11} = b^2c^2\sin^2 \alpha,$$

$$S_{22} = a^2c^2\sin^2 \beta,$$

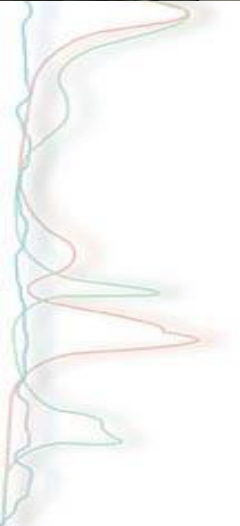
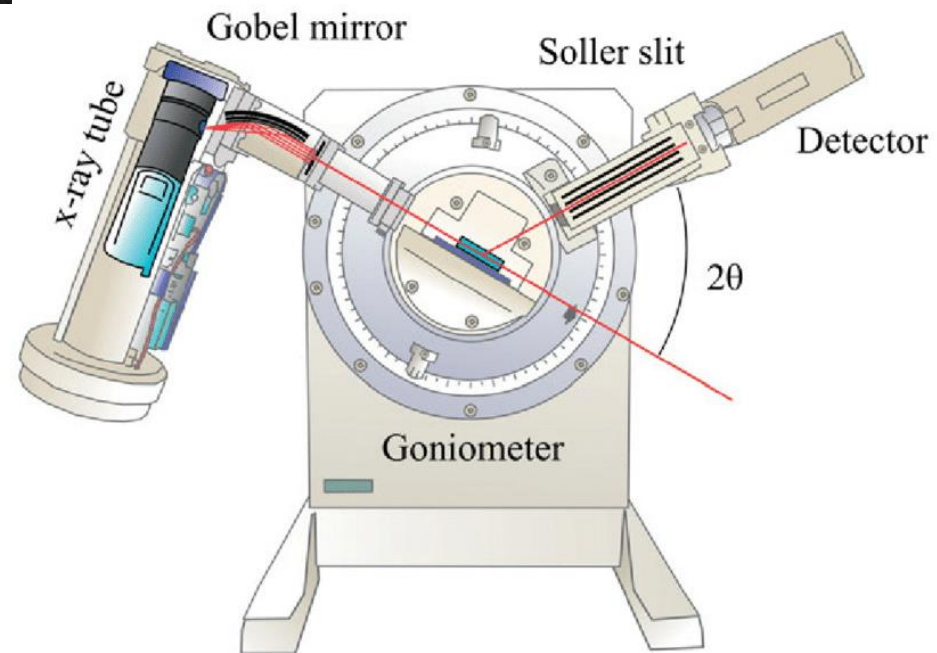
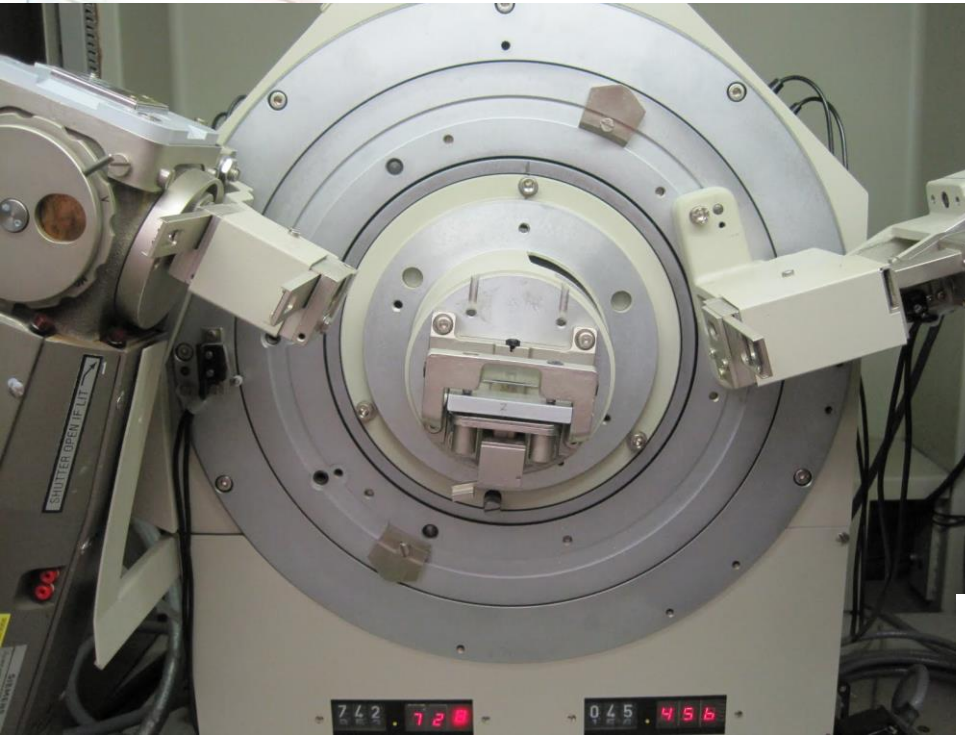
$$S_{33} = a^2b^2\sin^2 \gamma,$$

$$S_{12} = abc^2(\cos \alpha \cos \beta - \cos \gamma),$$

$$S_{23} = a^2bc(\cos \beta \cos \gamma - \cos \alpha),$$

$$S_{13} = ab^2c(\cos \gamma \cos \alpha - \cos \beta).$$

Instrumentation





Instrumentation

X-ray Source Components

Components for the source include:

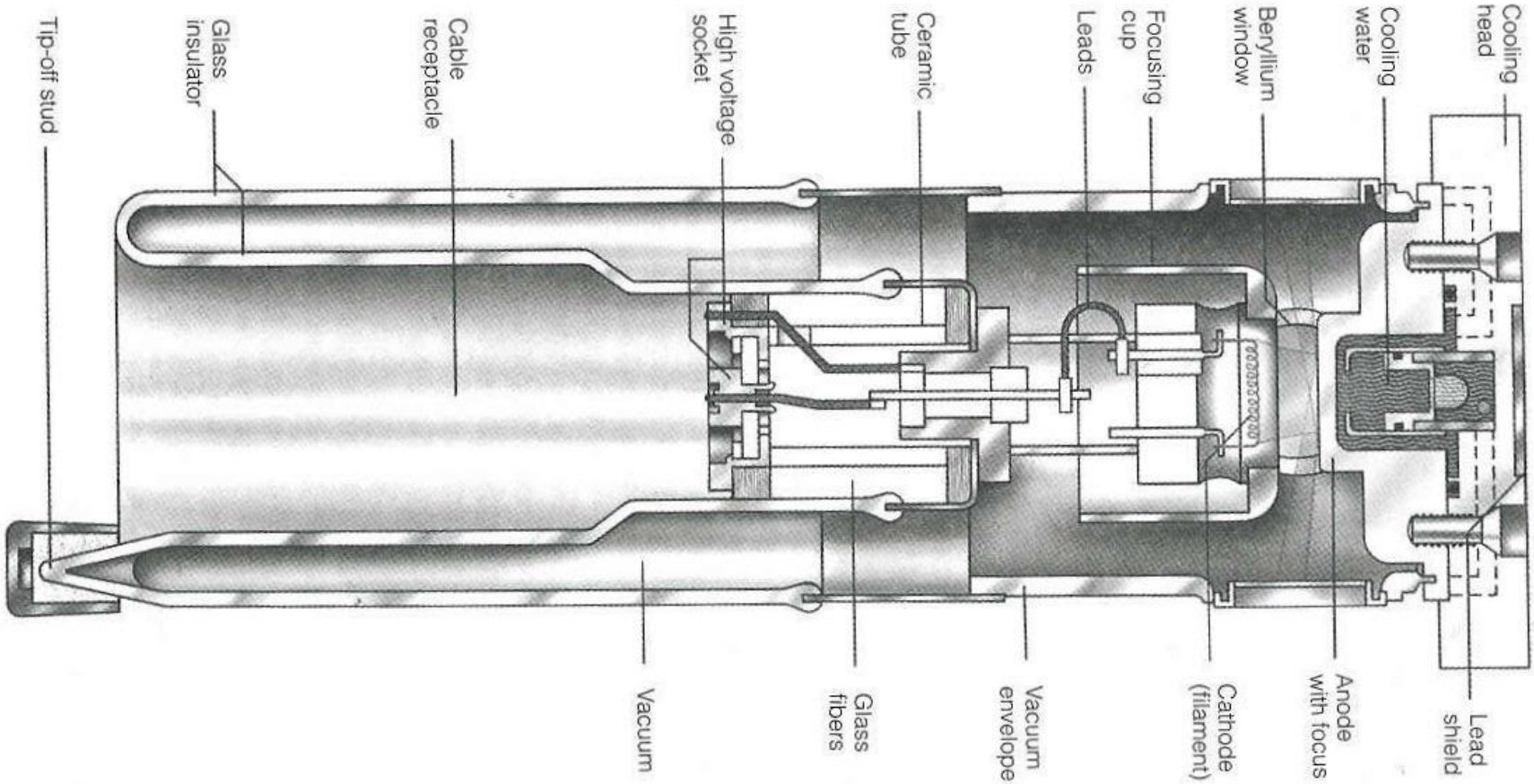
- Line voltage supply
- high-voltage generator
- x-ray tube

X-ray source requires

- high photon output
- high specific intensity
- selectable levels kV and mA
- stable output

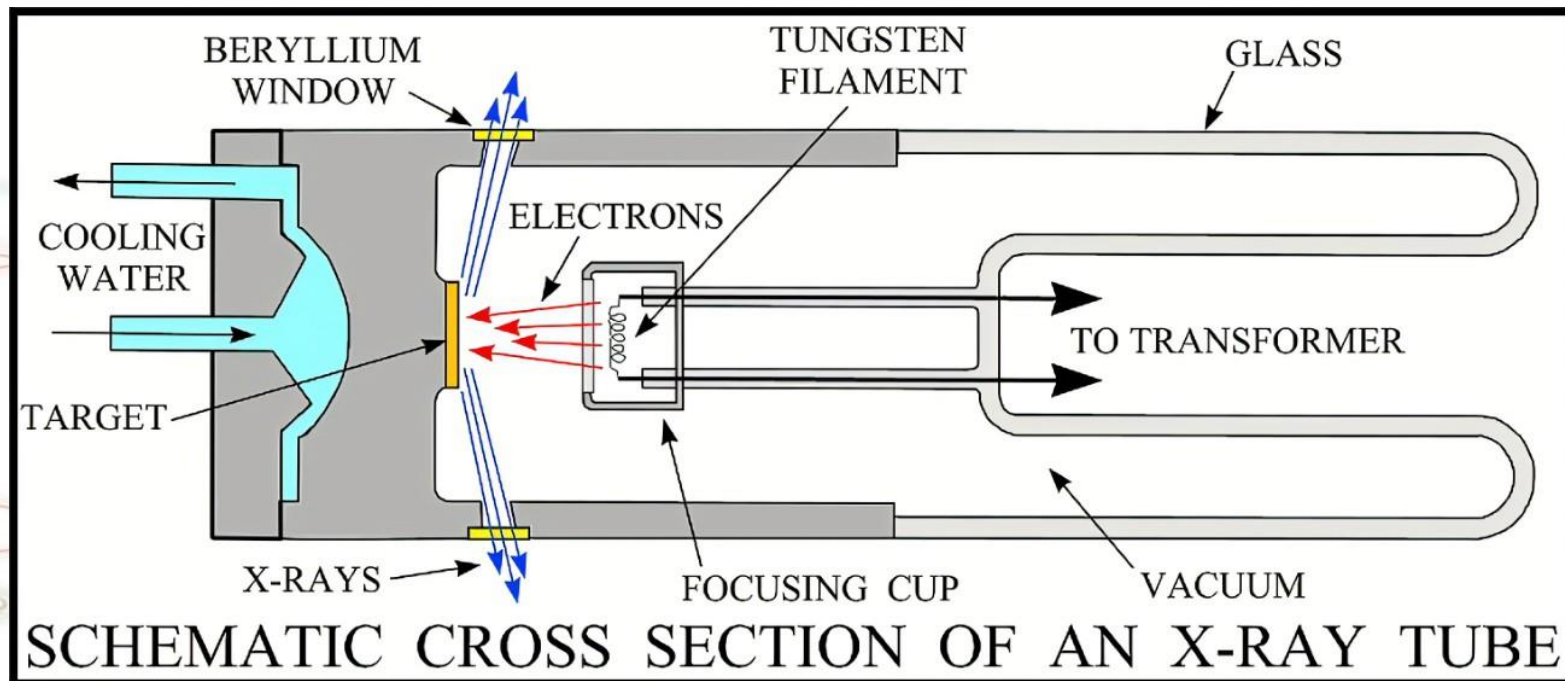
Instrumentation

X-ray Source Components



Instrumentation

X-ray Source Components



X-rays are produced whenever high-speed electrons collide with a metal target. A source of electrons – hot W filament, a high accelerating voltage between the cathode (W) and the anode and a metal target, *Cu*, Al, Mo, Mg. The anode is a water-cooled block of Cu containing desired target metal.

Instrumentation

X-ray Source Components - Cathode

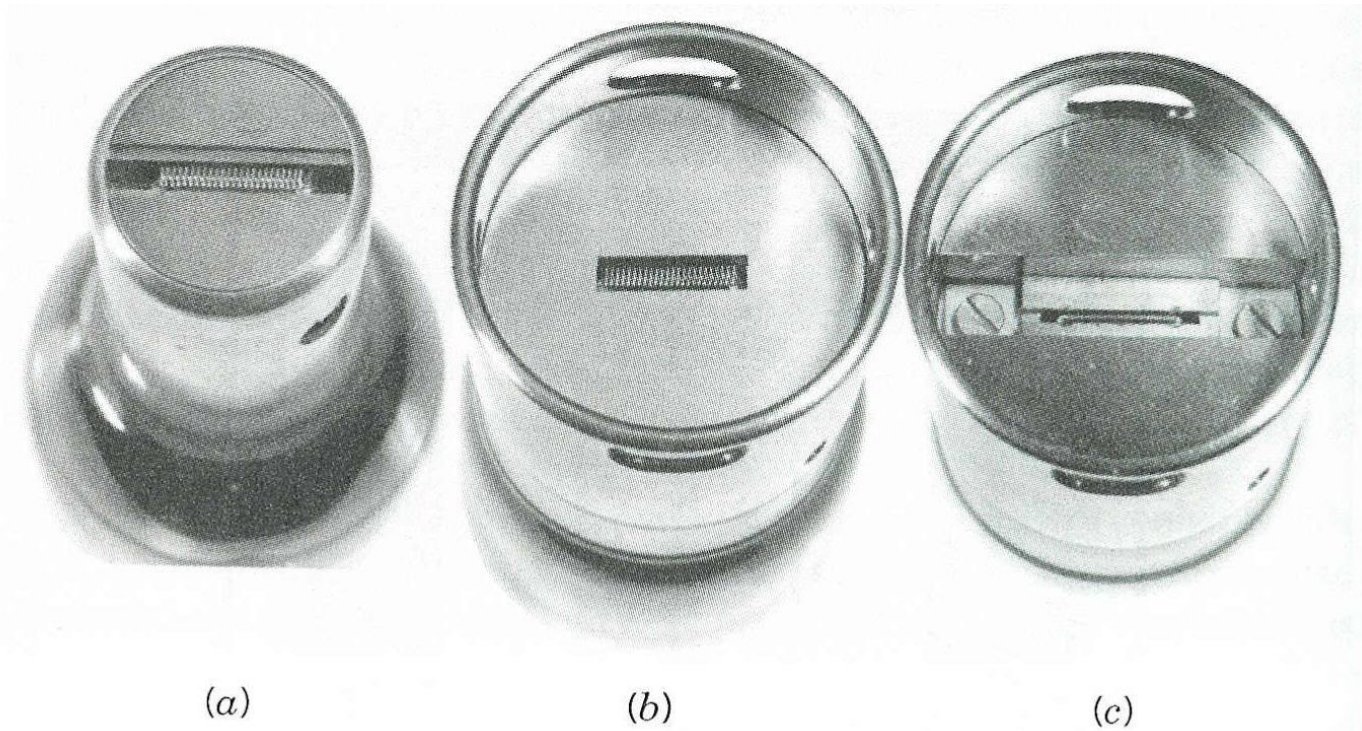


Figure 4.8. X-ray tube filament assemblies: (a) broad focus; (b) normal focus; (c) fine focus. Reprinted from R. Jenkins and J. L. de Vries, *An Introduction to Powder Diffractometry*, p. 19, Fig. 23. Copyright © 1977, N. V. Philips, Eindhoven, The Netherlands.

Instrumentation

X-ray Source Components - Anode

Anode is a cooled block of metal – Cu, Ni, Mo, etc...

The specific loading (W/mm²) of the anode is rated for tubes.

| <u>Tube type</u> | <u>Dimensions (mm)</u> | <u>Loading (kW)</u> | <u>SpecificLoading(W/mm²)</u> |
|-----------------------|------------------------|---------------------|--|
| Fine focus | 0.5 x 12 | 2.0 | 333 |
| Normal focus | 1.0 x 12 | 2.5 | 208 |
| Broad focus | 2.0 x 12 | 3.0 | 125 |
| Rotating Anode | 0.5 x 10 | 15.0 | 3000 |

Instrumentation

Optics

Includes a variety of slits, filters, mirrors, monochromators

Purpose: to reduce stray radiation, produce x-ray spectra which display diffraction from a single wavelength. (each unique d-spacing will diffract different wavelengths at different angles).

Instrumentation

Optics

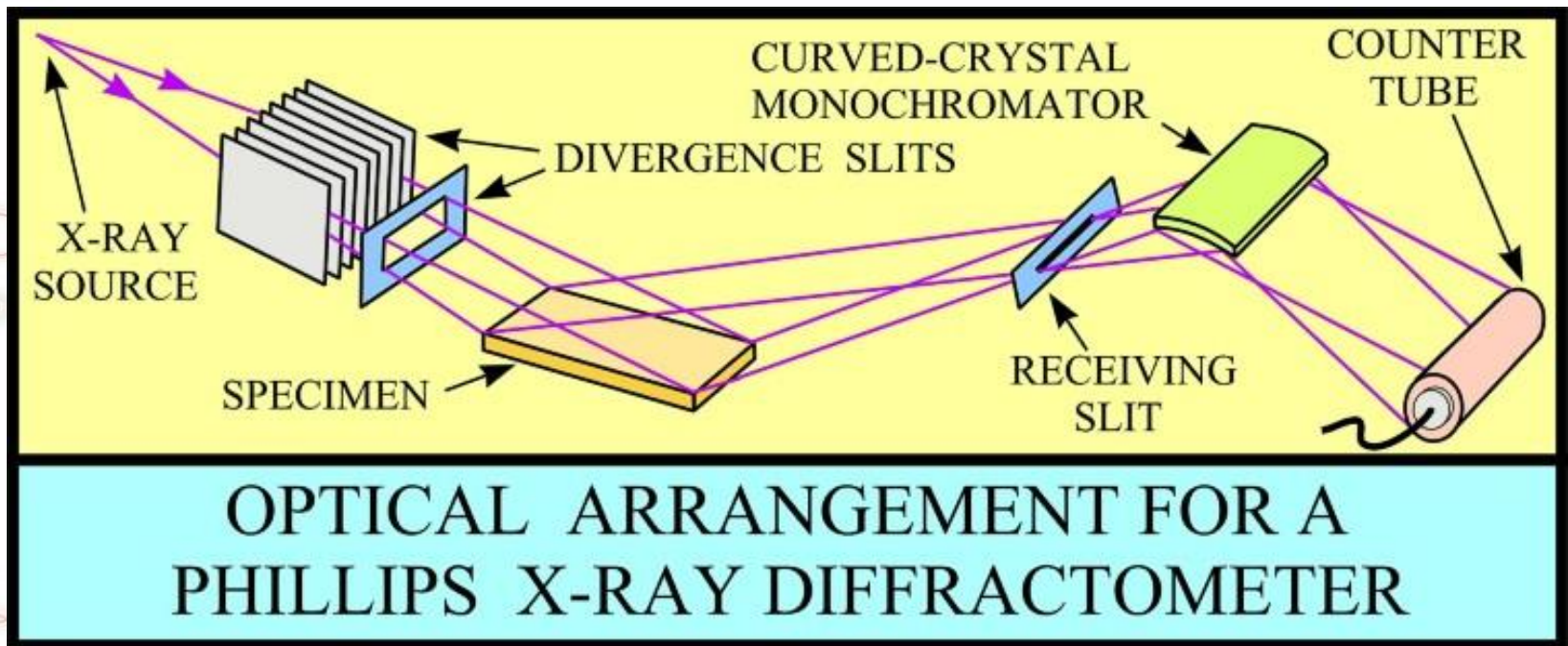
Besides the wanted x-ray wavelengths, a diffraction pattern is also made up of scatter and fluorescence.

| <u>Source</u> | <u>Result</u> |
|-----------------------------------|----------------------------|
| Diffraction of required λ | Wanted peaks |
| Diffraction of other λ 's | Unwanted peaks |
| Coherent scatter from sample | General background |
| Incoherent scatter from sample | General background |
| Scatter from sample support | Extra low-angle background |
| Fluorescence from sample | General background |

Instrumentation

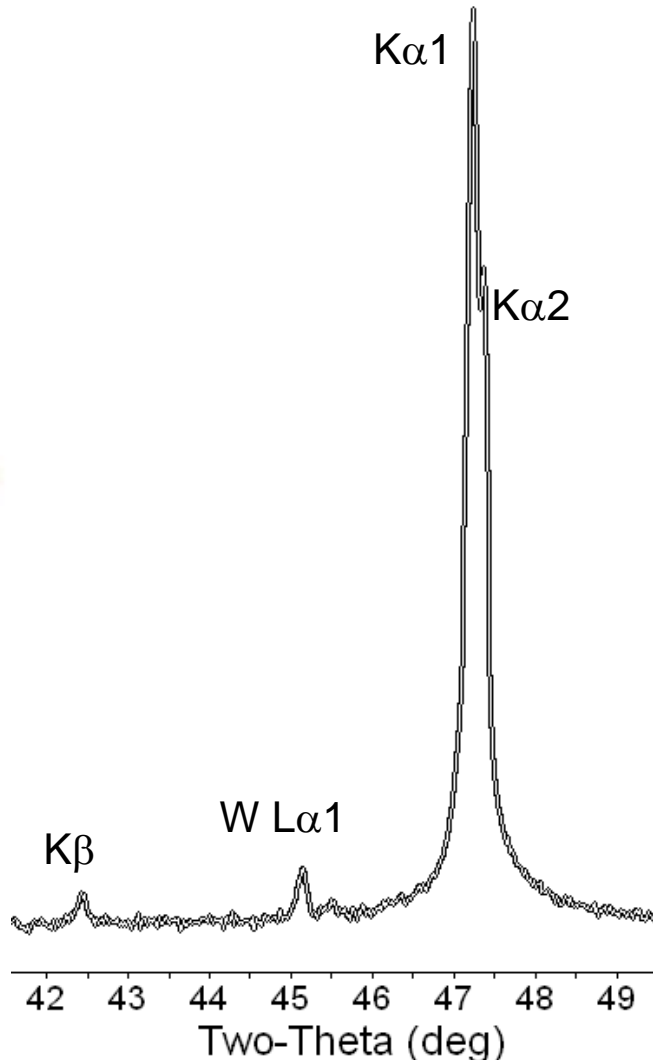
Optics

The fluorescence background is relatively constant, since it is not diffracted and angle independent, a fixed divergent slit can decrease this problem.



Instrumentation

Spectral Contamination in Diffraction Patterns

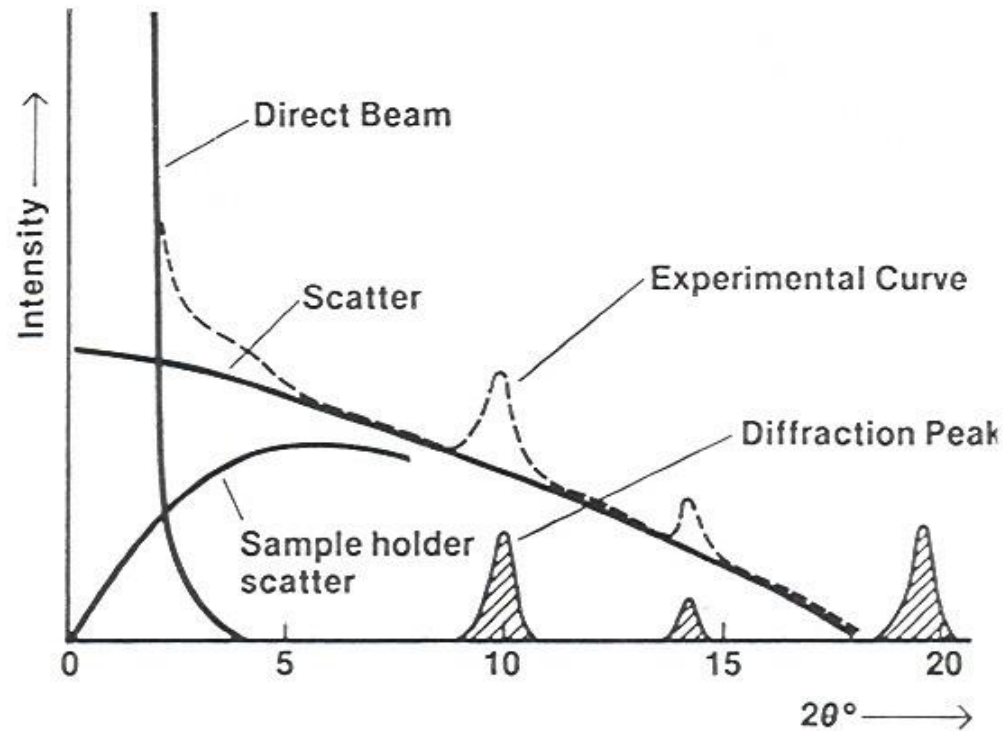


Additional lines in the diffraction pattern occur because of an impure source. These can include $Fe K\alpha$, $W\alpha$, and $W LB$.

Instrumentation

Optics

The low-angle region (0 to 20°) is especially difficult to clean up. The sample holder interferes at these angles.



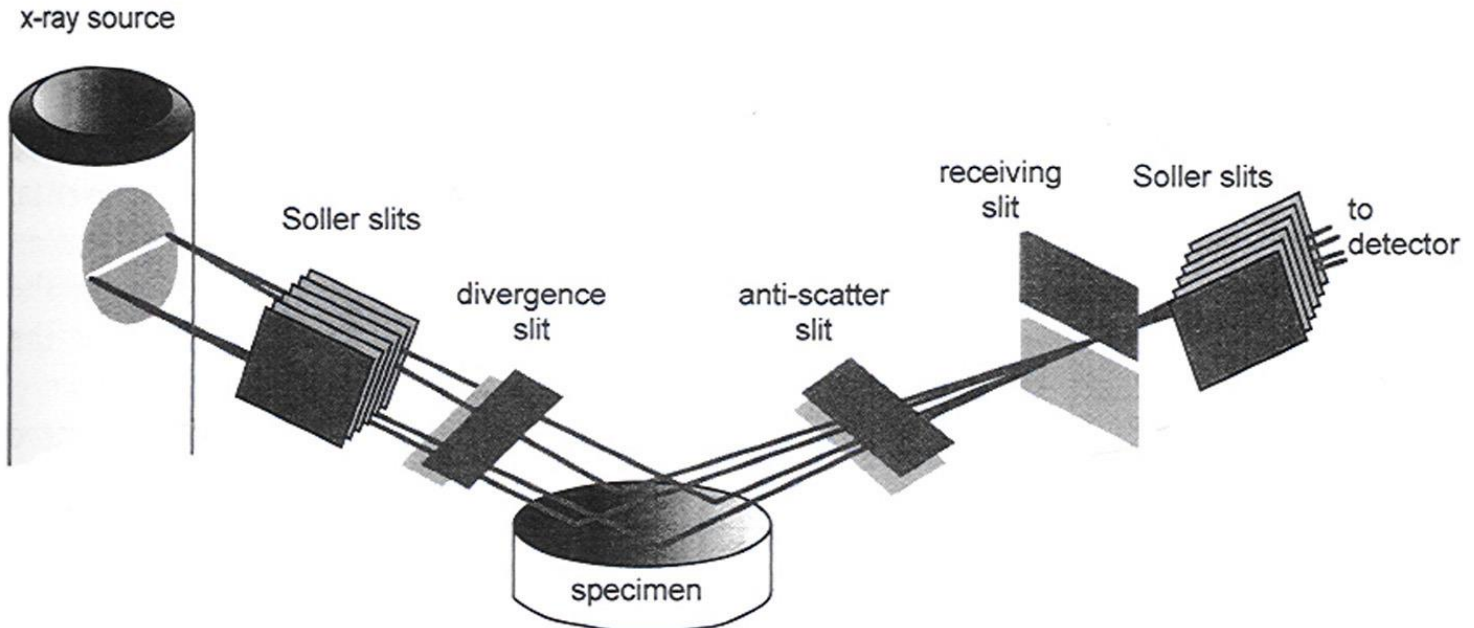
Instrumentation

Optics

Various Slits

The x-ray radiation passes through a series of slits on both the source side and the detector side.

These slit width can be varied depending on the sample and x-ray scan parameters.



Instrumentation

Optics

Various Slits

Soller slits – are a series of closely spaced metal plates which are parallel to each other.

These plates collimate (make parallel) the incident beam. The slits are typically 30 mm long and 0.05 mm thick, the distance between the plates is about 0.5 mm.

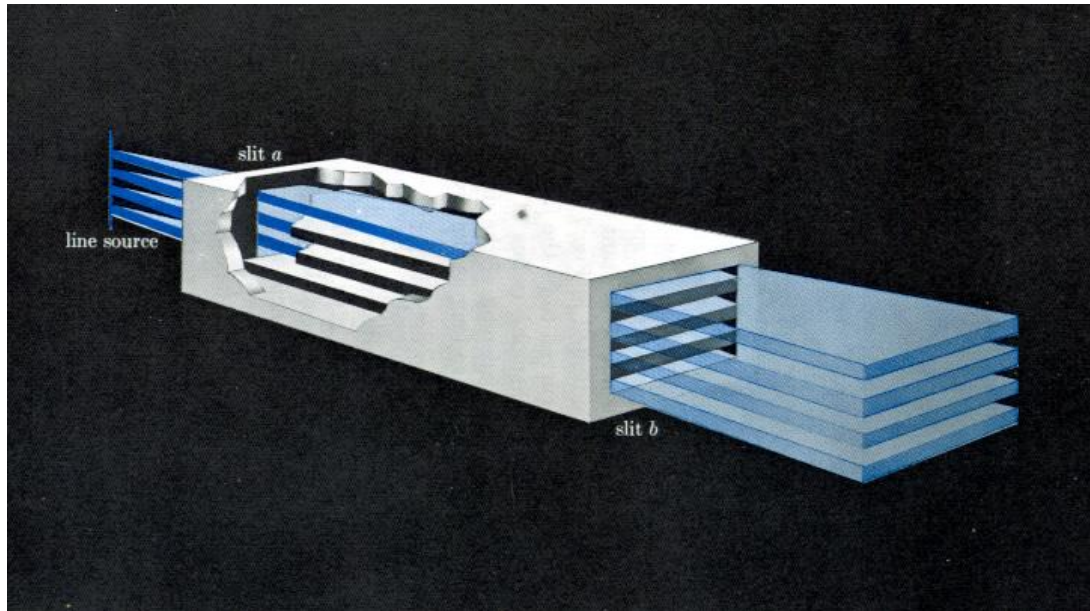
These plates are made up of a high atomic number element such as Mo or Ta.

Soller slits – sometimes a second set is present before the monochromator.

Instrumentation

Optics

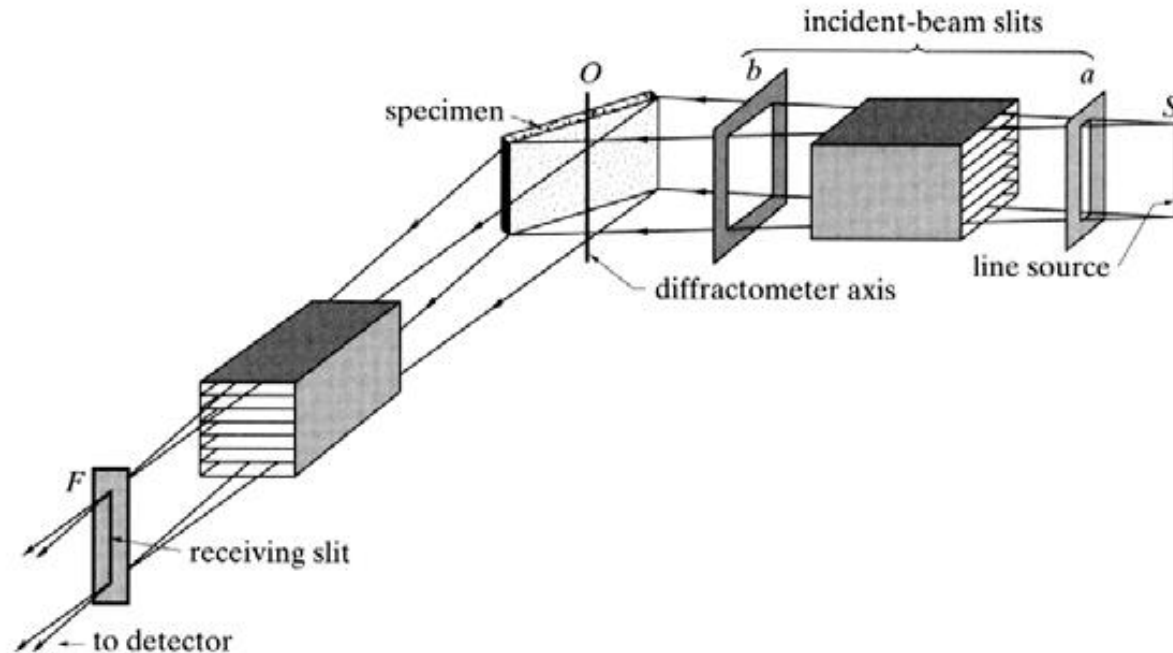
Soller slits - take a line source of radiation and slice it into smaller, parallel beams. This reduces axial divergence of the beam.



Instrumentation

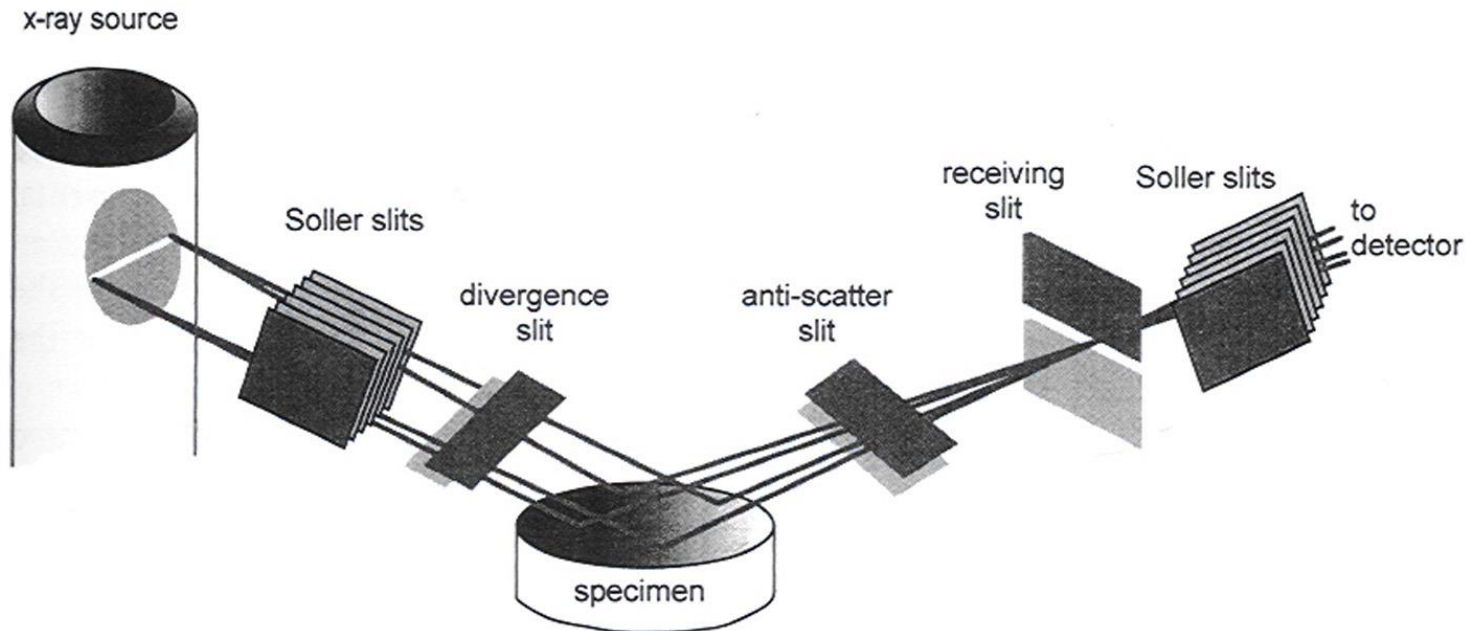
Optics

Soller slits - The sizes of the slits determine the intensity of the peaks measured in the diffraction pattern and also their shapes. Narrow slits reduce the intensity but they also produce sharper peaks.



Instrumentation

Various Slits



Divergence slit – defines the width of the incident beam.

Divergence slit - limits the vertical divergence of the x-ray beam, to irradiate as much of sample as possible while avoiding the sample support.

Instrumentation

Optics

Divergence slit – slits are fitted in the incident beam path to control the amount (length) of the sample that is irradiated by the incident x-ray beam. Can be fixed or variable.

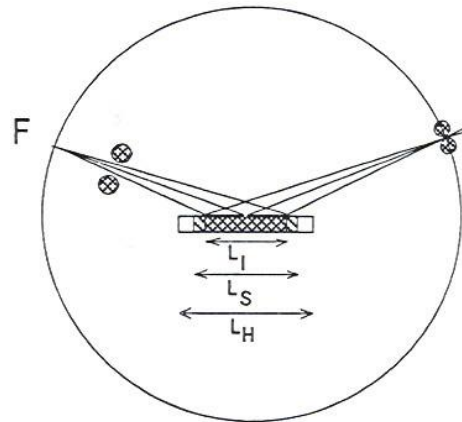


Instrumentation

Optics

Divergence slit

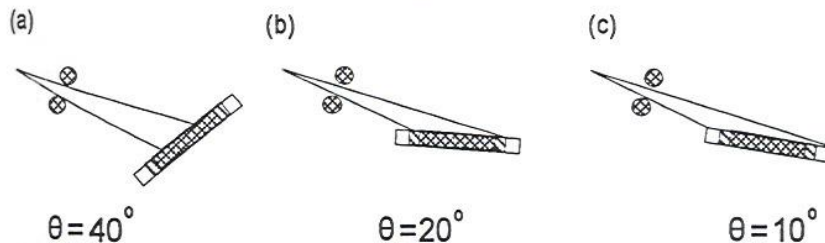
If the divergence slit is fixed, the irradiation area of the sample will change with angle.



At 40° , only the sample is irradiated

At 20° , sample and some support is irradiated, increasing background.

At 10° , larger part of sample holder is irradiated, increase background.



Instrumentation

Optics

Divergence slit

Too wide a divergence slit, or too small a sample holder, can lead to a small scatter peak (ghost) at $4-5^\circ 2\theta$, which can be confused with a clay peak.

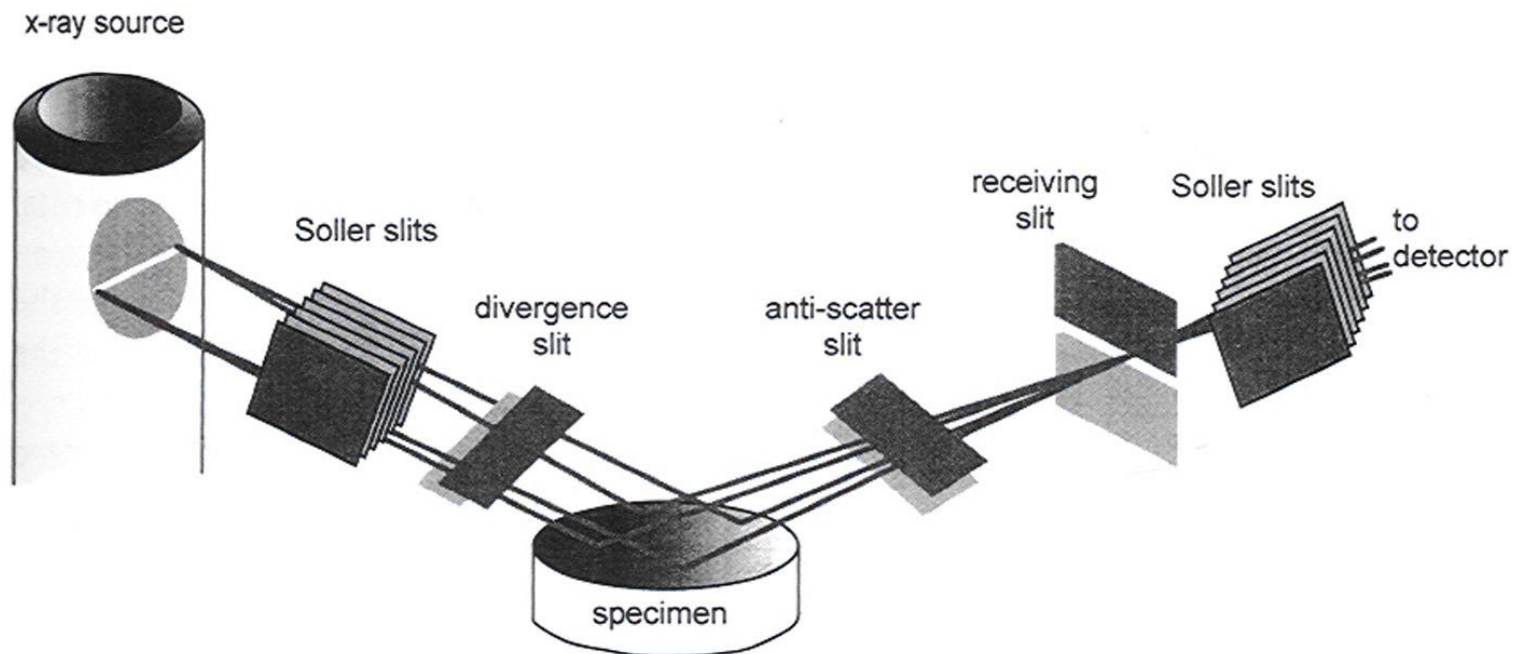
So the choice of divergence slit is critical, especially at low angles.

Instrumentation

Optics

Antiscatter slits – reduces background radiation.

Antiscatter slits – The anti-scatter slit not only reduces the height divergence but also reduces diffusely scattered x-rays which are due to amorphous or air scattering. This results in a reduction in noise of the output.

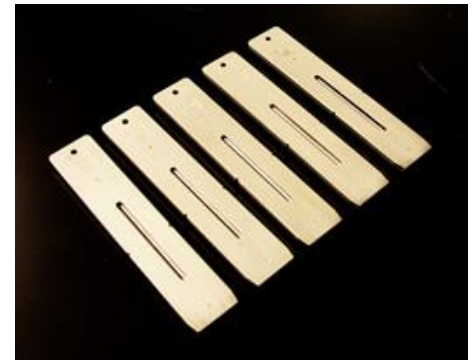
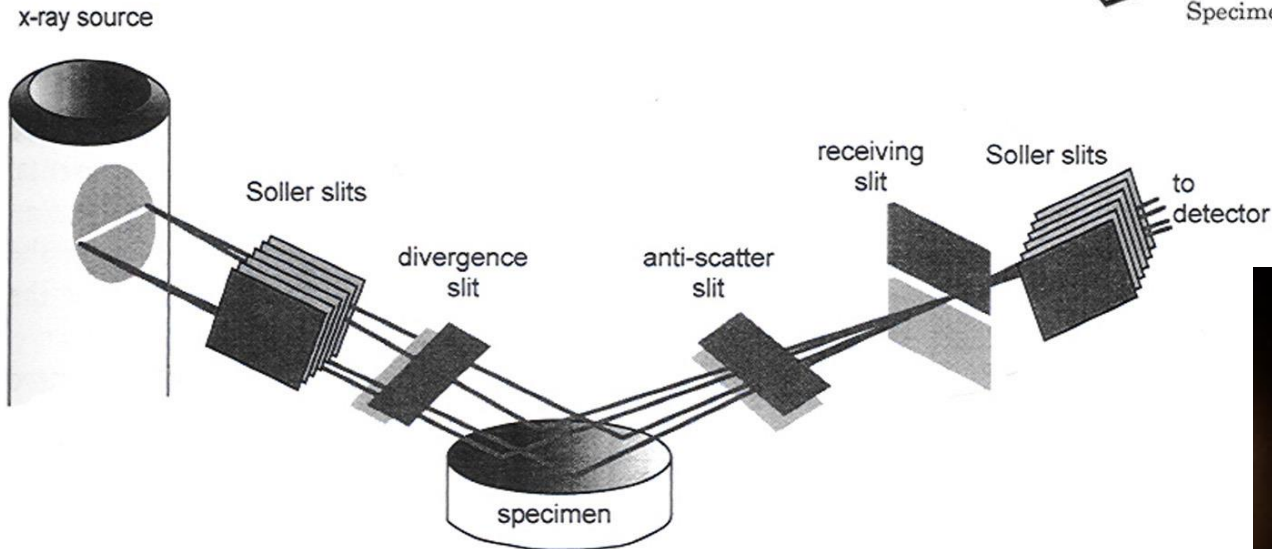
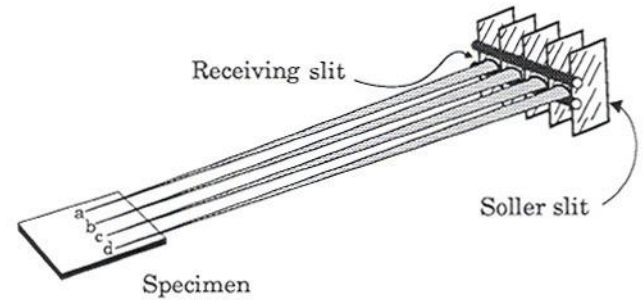


Instrumentation

Optics

Receiving slit – defines the width of the diffracted beam.

The receiving slit size can be varied, and has a dramatic effect on the peak shape and intensity.



Instrumentation

Optics

Receiving slit

There is only one optimum size receiving slit. Want to choose the beam width close in size to the receiving slit for optimum intensity and resolution.

Receiving slit is smaller than the beam width - intensity is reduced with slight improvement of resolution.

Receiving slit is larger than the beam width - slight increase in intensity but very poor resolution.

Instrumentation

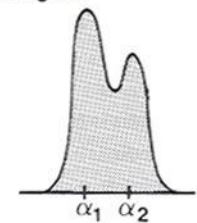
Optics

Receiving slit

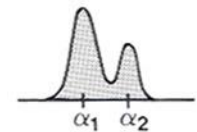
Receiving slit is smaller than the beam width - intensity is reduced with slight improvement of resolution.

Receiving slit is larger than the beam width - slight increase in intensity but very poor resolution.

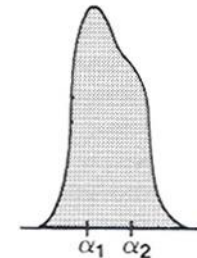
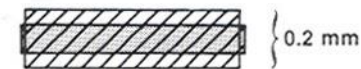
(a) Beam is exactly coincident with receiving slit



(b) Receiving slit smaller than image



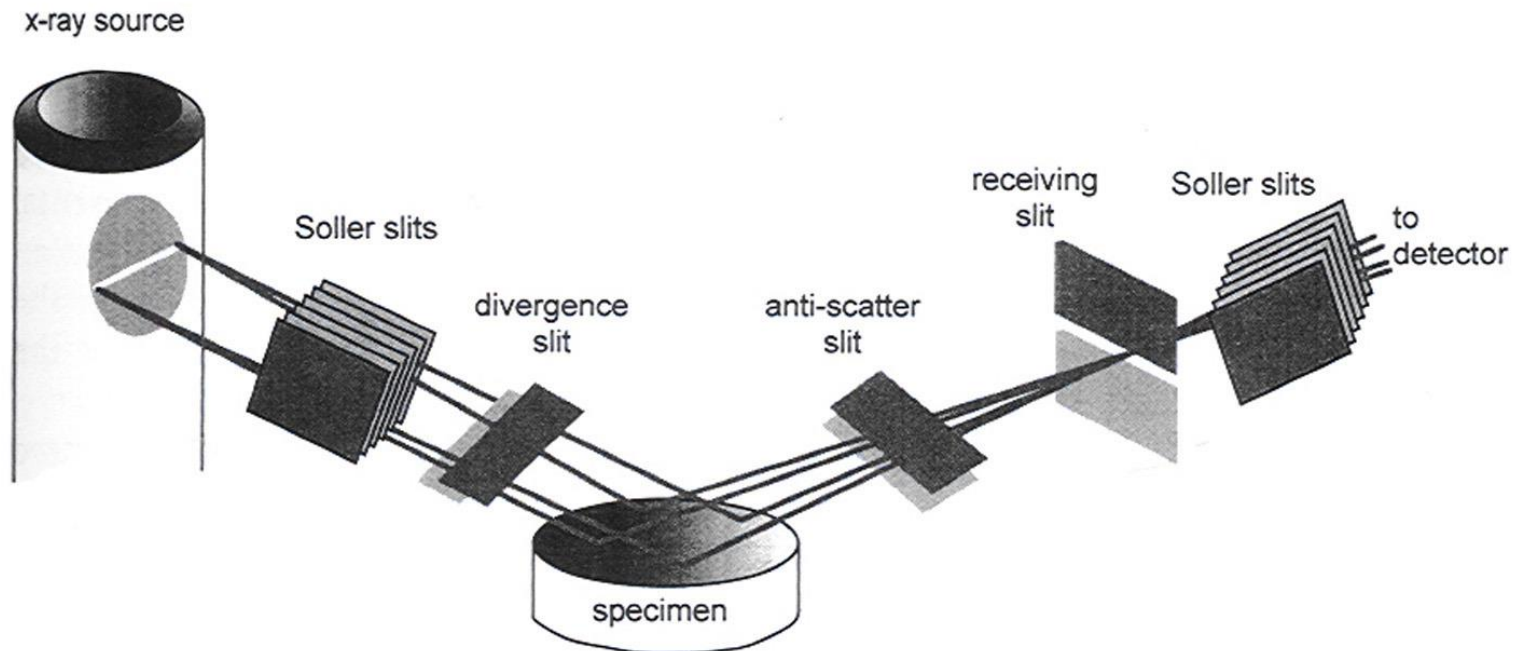
(c) Receiving slit larger than image



Instrumentation

Optics

Detector slit - a slit situated right before the detector. This slit is part of the monochromator system and can be adjusted to allow the α doublet to pass through the slit, while excluding the β radiation.

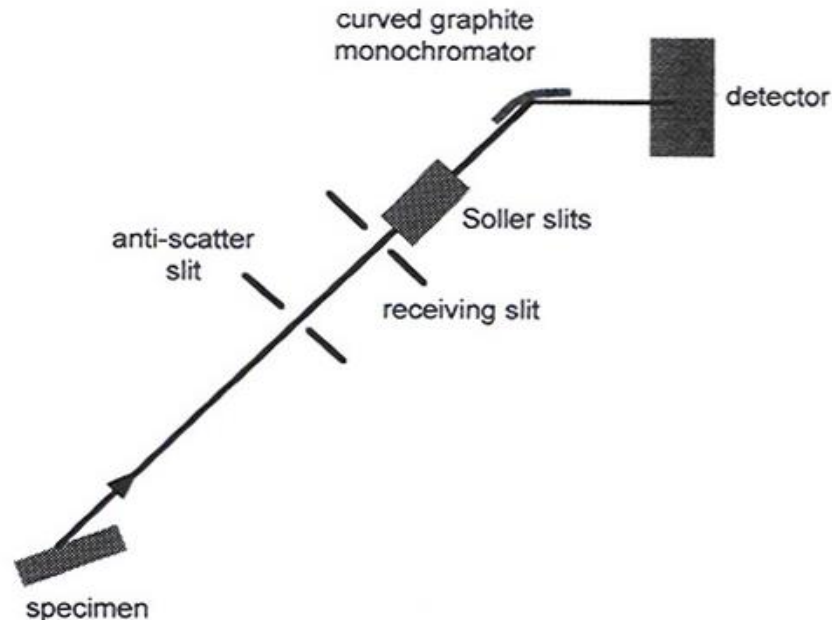


Instrumentation

Optics

Monochromators

Curved crystal monochromators provide monochromatic radiation with low background, and furnish high intensity (compared to plane) and high resolving power. Materials used as crystals are: mica, gypsum, quartz, graphite, Si, Ge, LiCl.

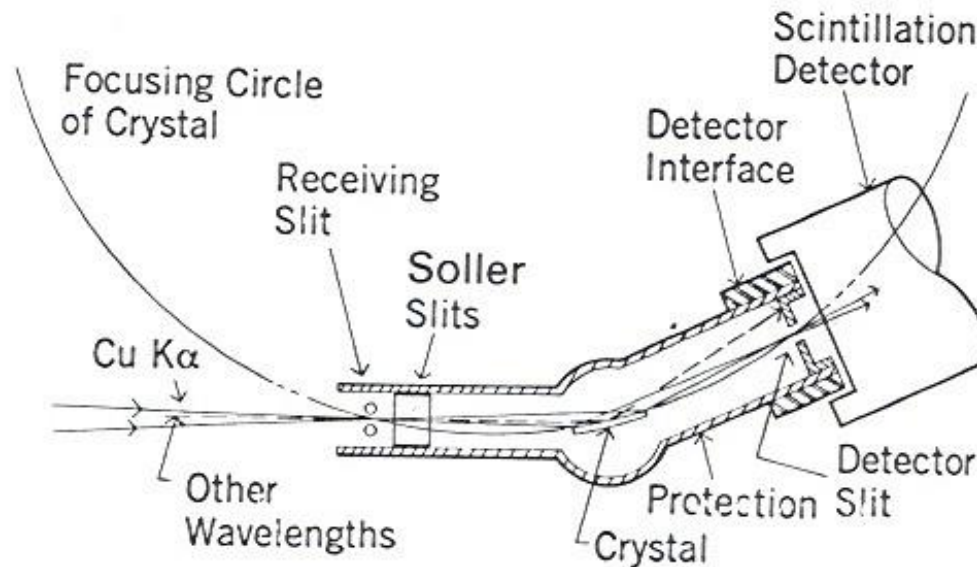


Instrumentation

Optics

Monochromators

Diffracted beam monochromator is made up of: a receiving slit, with a single crystal behind that, the detector is set at an angle to collect the λ of interest diffracted by the crystal. The surface of the crystal, receiving slit, and detector slit all lie on the focusing circle of the monochromator.



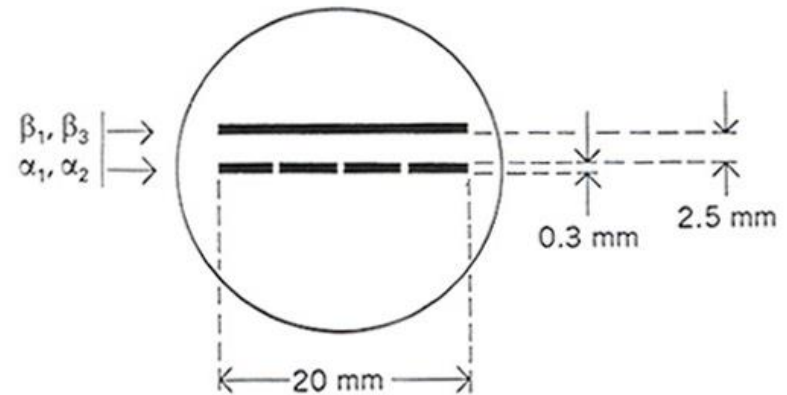
Instrumentation

Optics

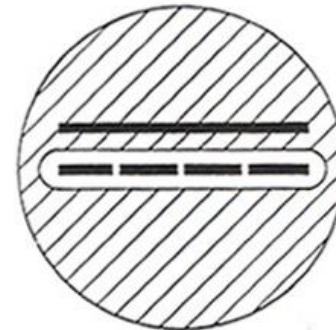
Monochromators

If the monochromator is correctly aligned, the α doublet is exactly at the center of the detector slit, while the β radiation would miss the slit.

Intensity Distribution at Detector Window



Intensity Distribution passed by Detector Slit

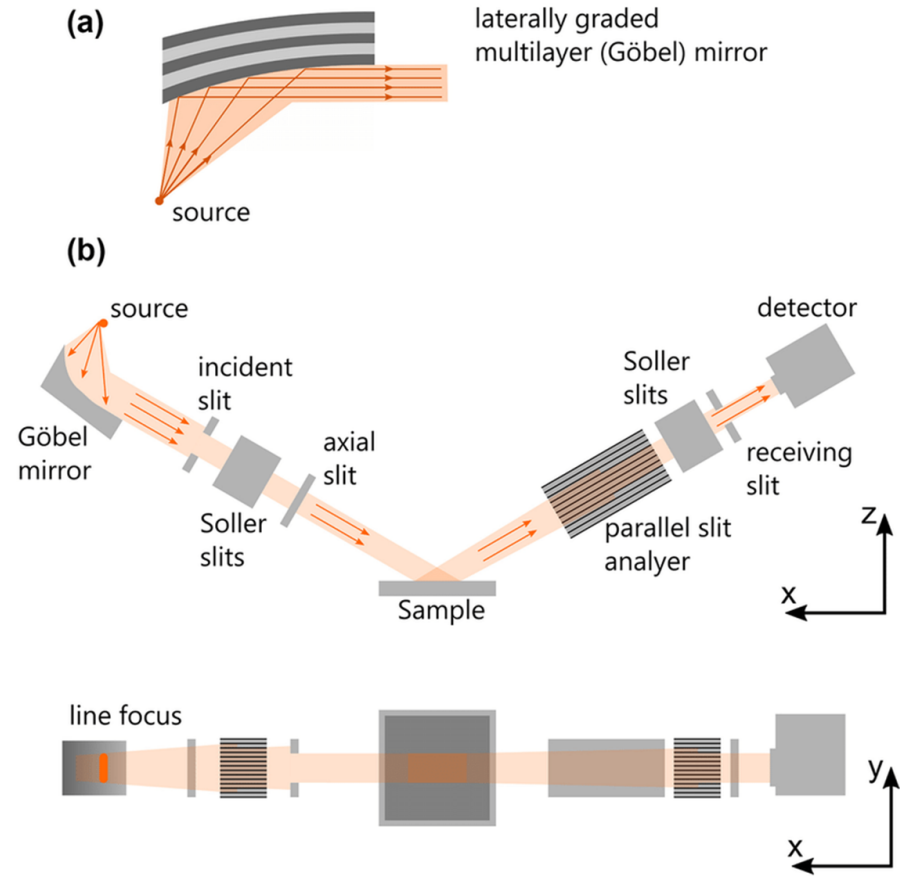


Instrumentation

Optics

Mirrors

If an instrument setup does not use a monochromator, it may use Gobel mirrors instead and have the ability for a parallel beam (PB) setup.

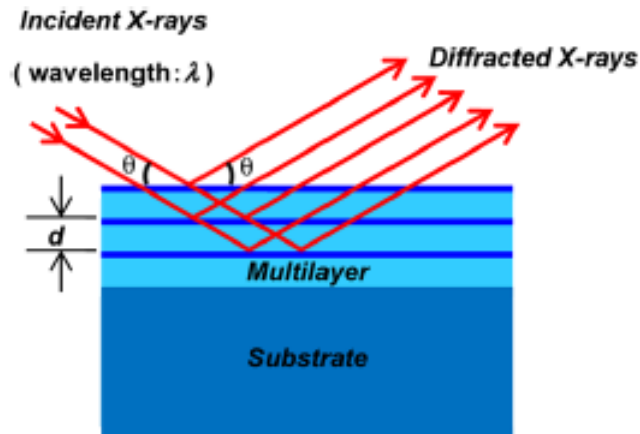


Instrumentation

Optics

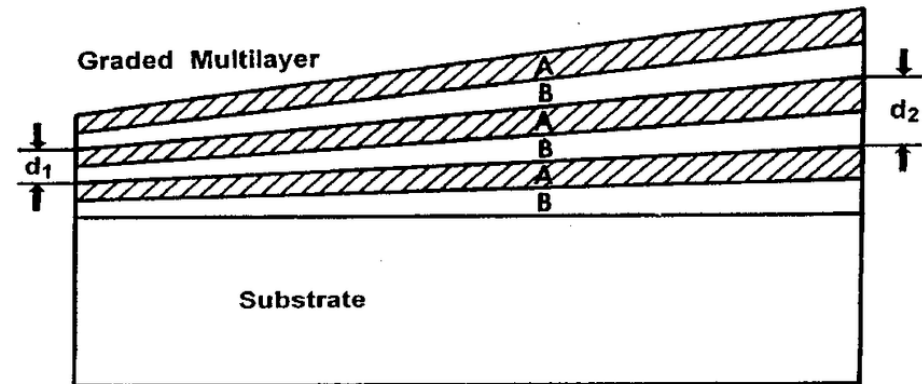
Mirrors

Gobel mirrors are made of a multilayer film, where the periodic thickness of the multilayer continuously changes along a parabolic surface.



$$\text{Bragg equation: } 2d \sin\theta = n\lambda$$

(d : interplanar spacing, θ : incident angle,
 n : integer, λ : wavelength)



Instrumentation

Optics

Mirrors

Table 1. Comparison of monochromatization methods.

| Monochromatic method | $K\beta/K\alpha$ intensity ratio | TDI measurement |
|-----------------------|----------------------------------|-----------------|
| $K\beta$ filter | Approx. 1% | Applicable |
| CBO- α | Below 0.2% | Applicable |
| Counter monochromator | Approx. 0.004% | N/A |

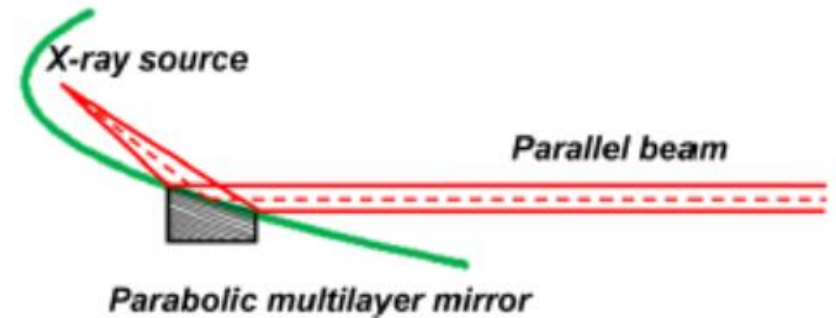
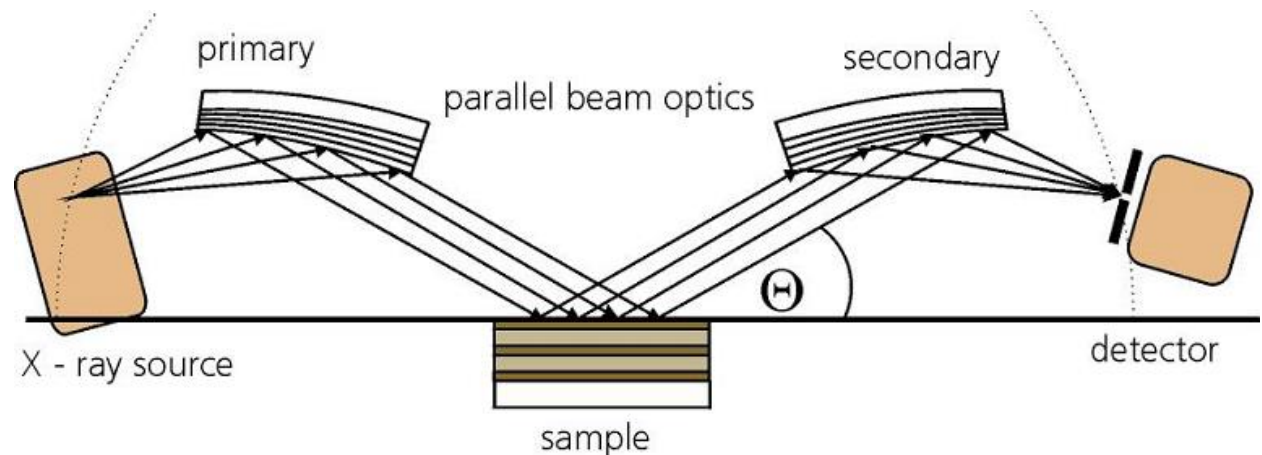


Fig. 2. Parabolic multilayer mirror.





Instrumentation

Properties of Detectors

- linearity or range (x-ray photons)
- quantum counting efficiency
- speed
- energy proportionality
- resolution
- sensitivity



Instrumentation

Detectors

Quantum counting efficiency – efficiency of the detector in collecting radiation. Ideal efficiency, $I \sim I_0$, for characteristic photons.

Speed

Dead time (τ) – time required for the detector to collect a photon, convert it to a pulse, and count the pulse.

Instrumentation

Detectors

Two types of dead time

paralyzable (nonextending) – complete saturation of the detector, causing detector to stop working. (usually over 100,000 counts/s)

nonparalyzable (extending) – increasing loss in counts with increasing count rate, but does not saturate.

Instrumentation

Detectors

Energy proportionality – when the size of the output pulse, V , is proportional to the energy, E , of the incident x-ray photon.

Resolution – measure of detectors ability to resolve two x-ray photons of differing energy.

$K\alpha$ and $K\beta$ energies of copper, are at 8.041 and 8.904 keV, respectively.

$K\alpha_1=8.047$ keV and $K\alpha_2=8.027$ keV.



Instrumentation

Detectors

Sensitivity – ability to detect low intensity levels.

Count rate - y axis – counts per second

Instrumentation

Detectors

Point detectors (0-D)

Scintillation detector
(NaI, YAP)

Gas proportional counter

Si(Li) solid state detector

Ge solid state detector

Silicon pin diode

Silicon drift detector

Ionization chamber

Linear detectors (1-D)

Gas proportional counter

Gas detector

Linear CCD

Micro-strip silicon
detector

Image plate detector (IP)

Photographic film

Area detectors (2-D)

Multi-wire proportional
counter

CCD-camera

Image plate detector (IP)

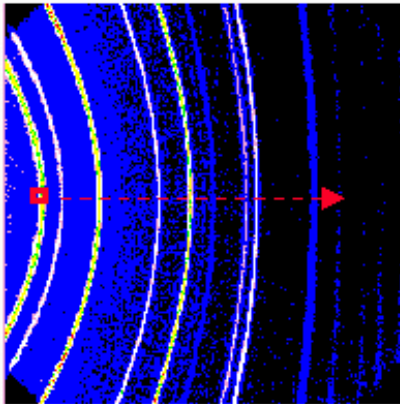
Photographic film



Instrumentation

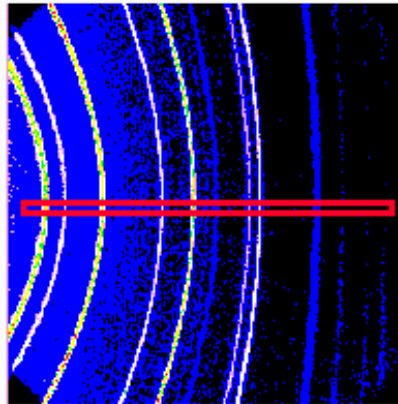
Detectors

scintillation
detector



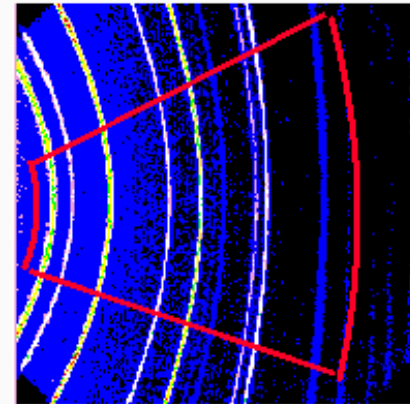
- small spot measured
- scan necessary
- long measuring time

PSD

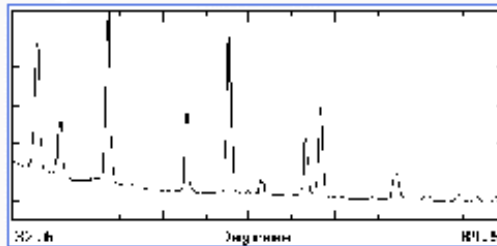


- large 2θ range measured simultaneously
- medium measuring time

GADDS



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



Instrumentation

Detectors

Some detectors depend on x-rays to ionize atoms (either as a gas or on a solid)

Proportional Detector

A metal tube (cathode) filled with a gas (i.e. Ar, Xe, or Kr) and contains a thin metal wire (anode) running down the center.

There is a constant potential difference between the cathode and anode.

X-rays enter the tube through a transparent window and are absorbed by a gas – typically Xe

The gas ejects a photoelectron and becomes ionize (an ion/electron pair of an electron and positive ion is produced)

Instrumentation

Proportional Detector

Ionized gas (+) moves toward the cathode (-)

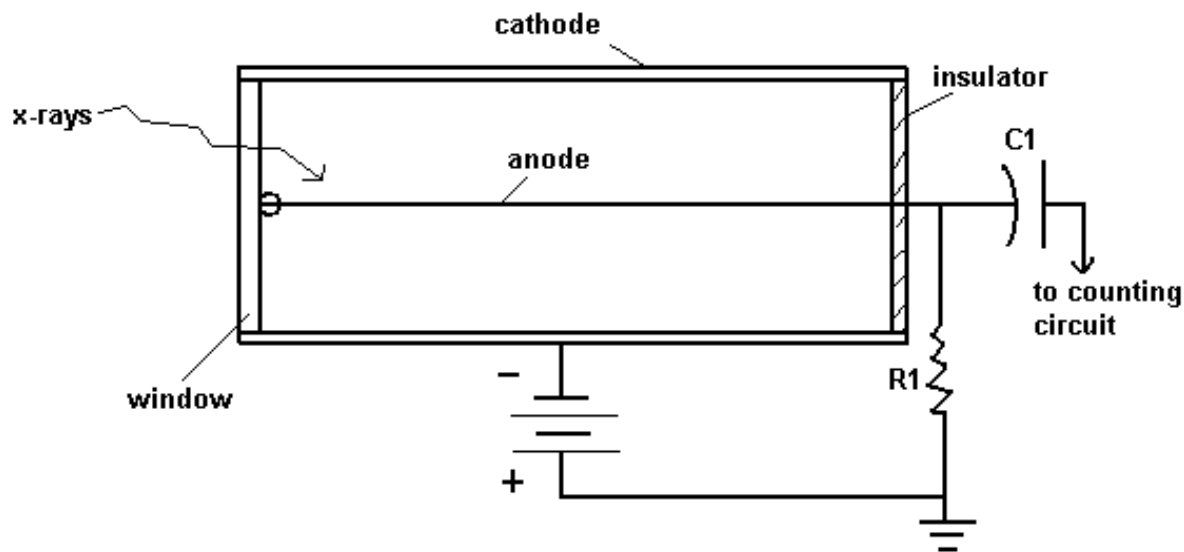
Electrons (-) move toward the anode (+)

A small current is measured and related to the x-ray intensity.

The ionization energy of the noble gas is ~20 - 30eV

For one Cu x-ray photon, the energy is 8.04 KeV

So ~270 electron-ion pairs are produced with $\text{CuK}\alpha$



Instrumentation

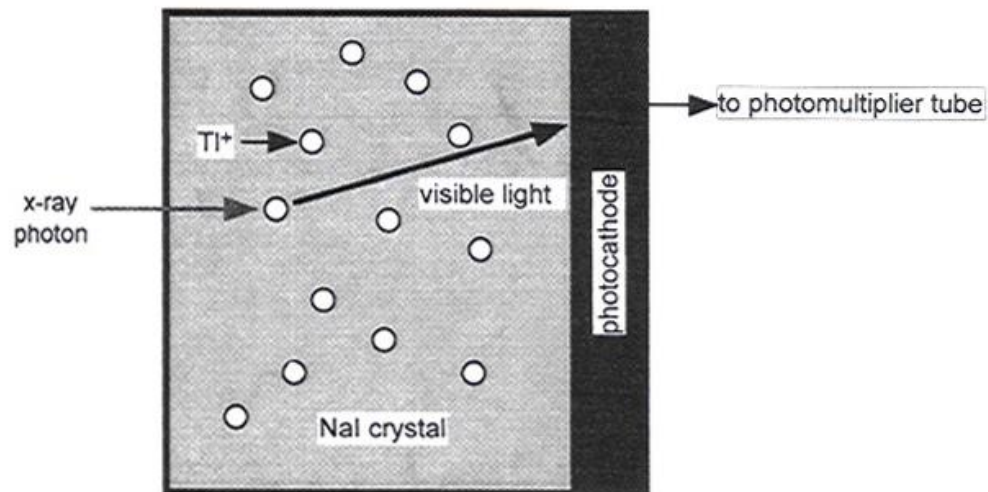
Scintillation Detector

Incident x-ray hits a crystal causing it to fluoresce.

The crystal is NaI doped with 1%TI (NaI/TI).

X-rays are absorbed by the crystal and raises electrons from the valence band to the conduction band in NaI.

These electrons transfer energy to the TI⁺ ion.



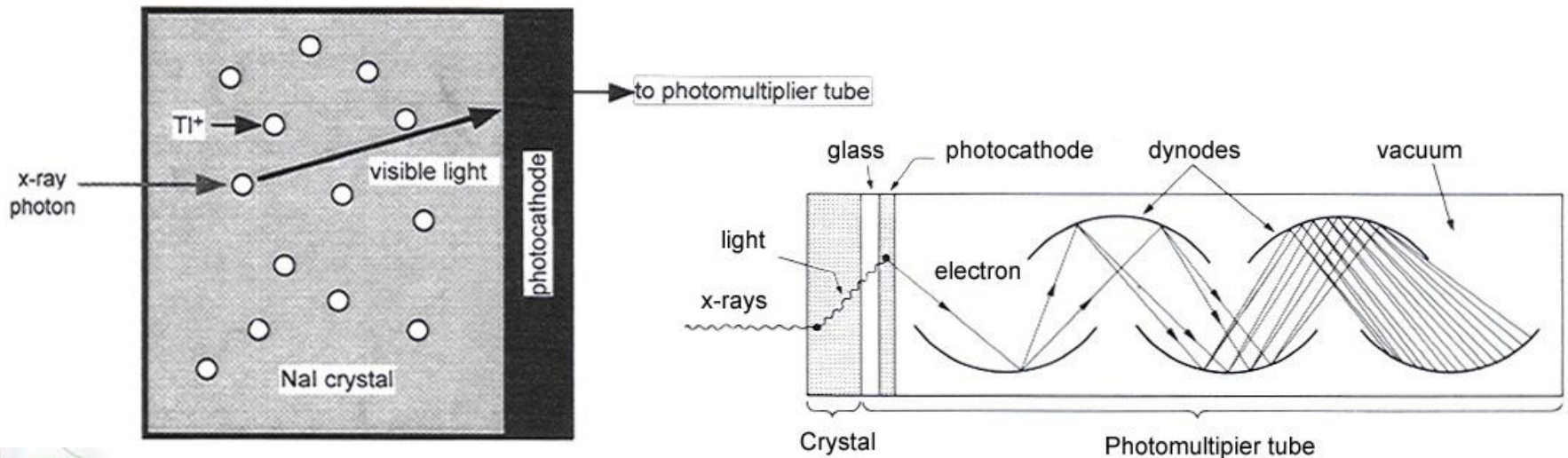
Instrumentation

Scintillation Detector

These electrons transfer energy to the Tl^+ ion.

The excited Tl^+ returns to ground state and emits light (fluoresce at $\lambda = 420$ nm).

A flash of light (scintillation) purple in color is produced in the crystal and is passed into a photomultiplier tube.



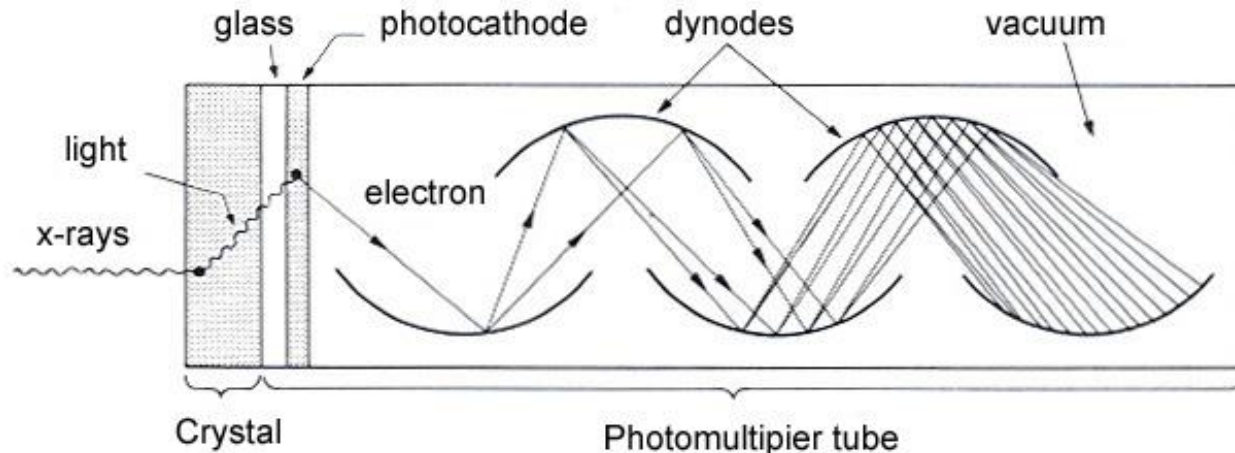
Instrumentation

Scintillation Detector

The photomultiplier tube is made up of a series (dynodes) of photocathodes.

The photocathodes are a photosensitive material made up of materials like cesium-antimony intermetallic compound.

Dynodes - coated with compounds such as BeO, GaP, and CsSb, which eject several electrons when subjected to the impact of a high-energy electron.



Instrumentation

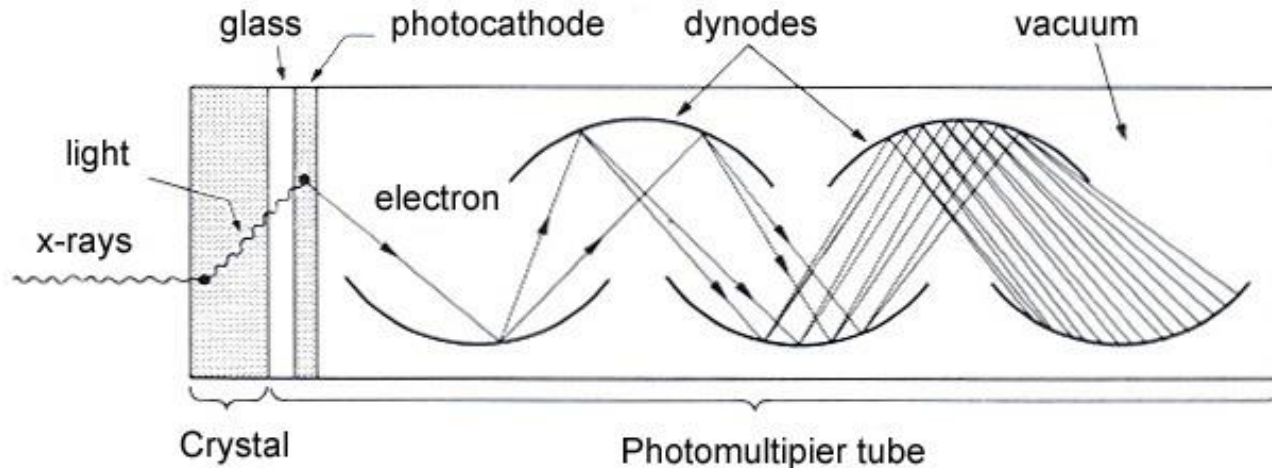
Scintillation Detector

Light strikes the 1st photocathode and electrons are ejected. These electrons are accelerated toward the next dynode by a potential difference (ΔV).

Each dynode is 100V more positive than the preceding one.

As electrons hit the next dynode, more electrons are produced (multiplication).

Last dynode is connected to a circuit.



Instrumentation

Scintillation Detector

The Cathode Quantum Efficiency (QE) equals

QE = average # of photoelectron emitted/# of incident photons.

Total Gain of the photomultiplier tube is:

$G = (f)^n$, where

f - secondary emission factor (range 3 - 50)

n - # of stages

If the Gain per dynode is ~5 (1 electron knocks out 4 to 5 electrons)

So with 10 dynodes, there is a multiplication factor of 5^{10} or 10^7 .

Instrumentation

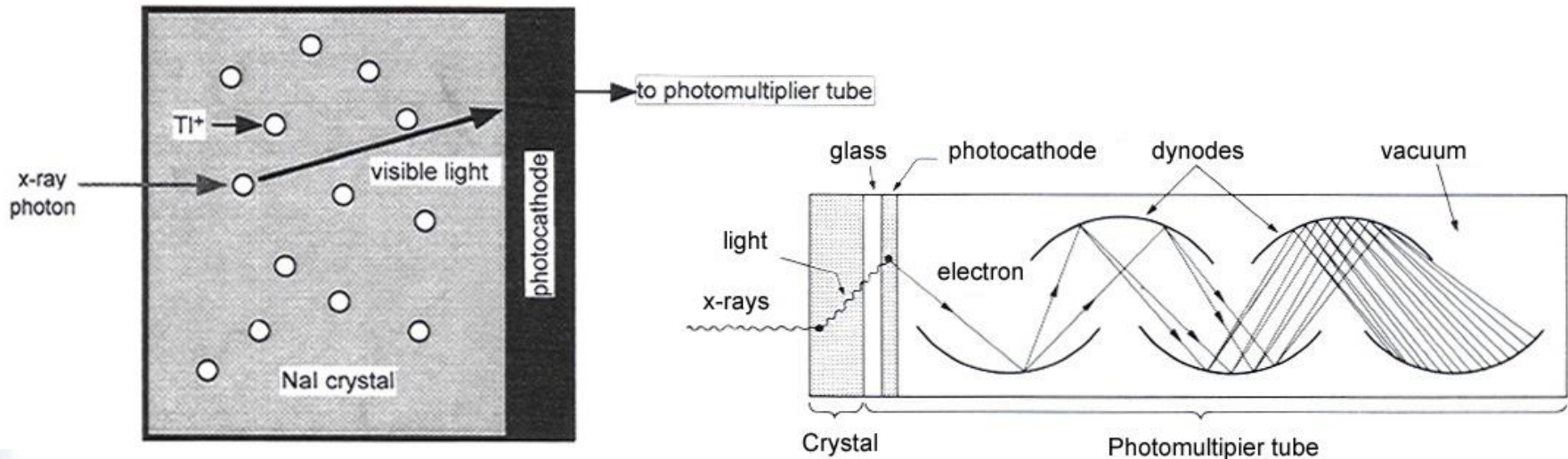
Scintillation Detector

This whole process takes less than a μsec .

So detector can handle rates of 10^5 counts/sec without loss.

Advantage - efficient detector $\sim 100\%$ and low dead time $\sim 0.1 \mu\text{s}$.

Disadvantage - energy resolution is not as good as the proportional detector or a solid state detector.



Application of Diffraction Data

XRD can be used for:

- Bravais lattice determination – phase determination (crystalline phases and orientation)**
- Lattice parameter determination**
- Determination of solvus line in phase diagrams (order-disorder transformation)**
- Long range order**
- Crystallite size and Strain**
- Temperature factor – thermal diffuse scattering (thermal expansion)**
- Thickness measurements of thin films and multilayers**

Application of Diffraction Data

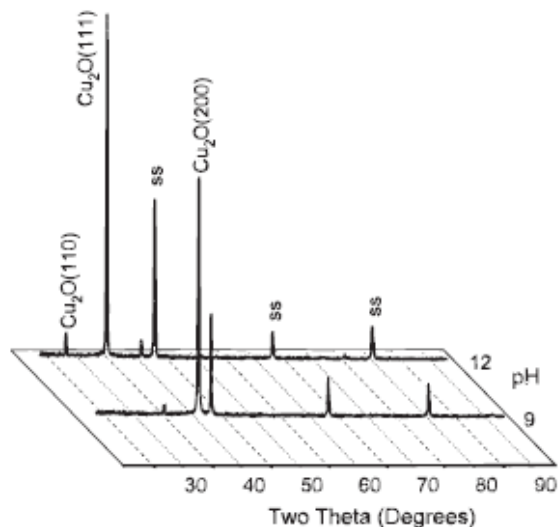


Figure 7. X-ray diffraction patterns of Cu₂O deposited from solutions of pH 9 and 12. The bath temperature is 65 °C and the applied current density is 0.8 mA/cm².

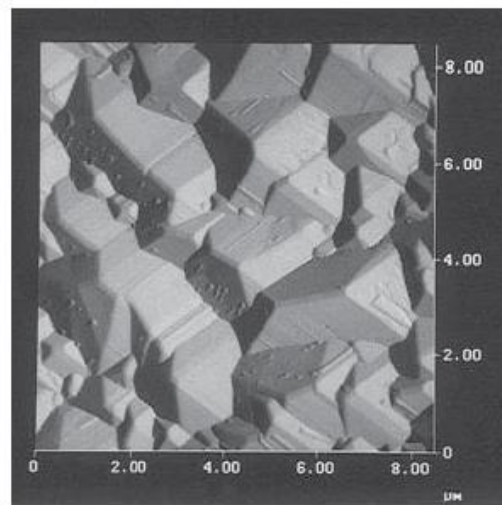


Figure 8. Top-view AFM image of a Cu₂O film deposited at pH 9, $E = -0.45$ V and 65 °C. AFM; z range = 2.5 μm, scan rate = 0.75 Hz.

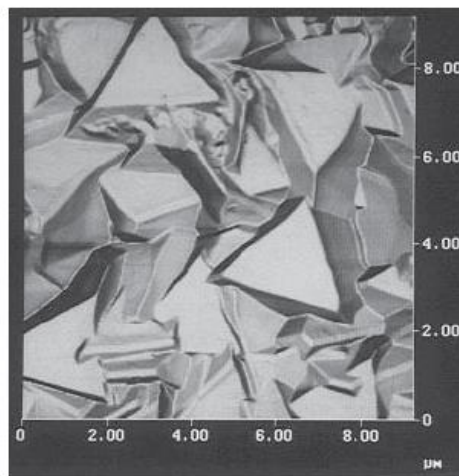
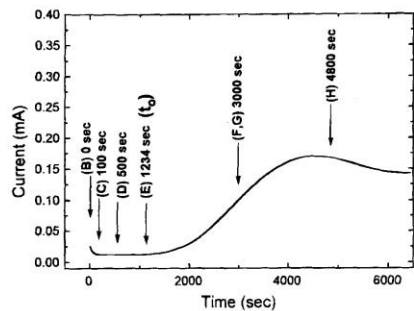
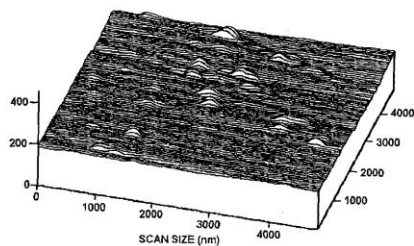


Figure 9. Top-view AFM image of a Cu₂O film deposited at pH 12, $E = -0.45$ V and 65 °C. AFM; z range = 4.2 μm, scan rate = 0.75 Hz.

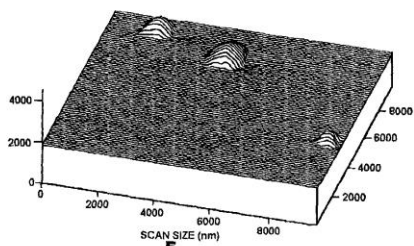
Application of Diffraction Data



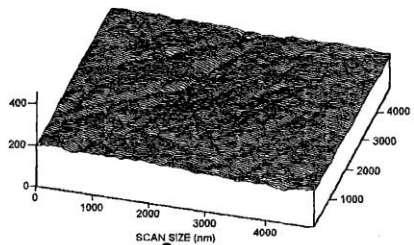
A



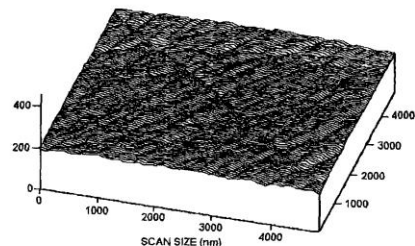
C



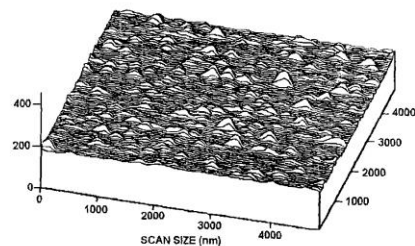
E



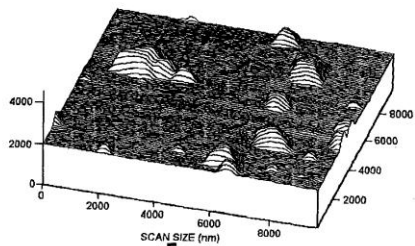
G



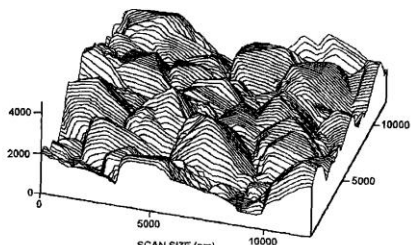
B



D



F



H

Fig. 12. Atomic force microscopy for several deposition times resulting from a potential step to an overpotential of 149 mV. (A) The location along the current-time transient for (B) 0 s, (C) 100 s, (D) 500 s, (E) 1234 s, (F) 3000 s, (G) 3000 s between nuclei over an amorphous surface and (H) 4800 s.

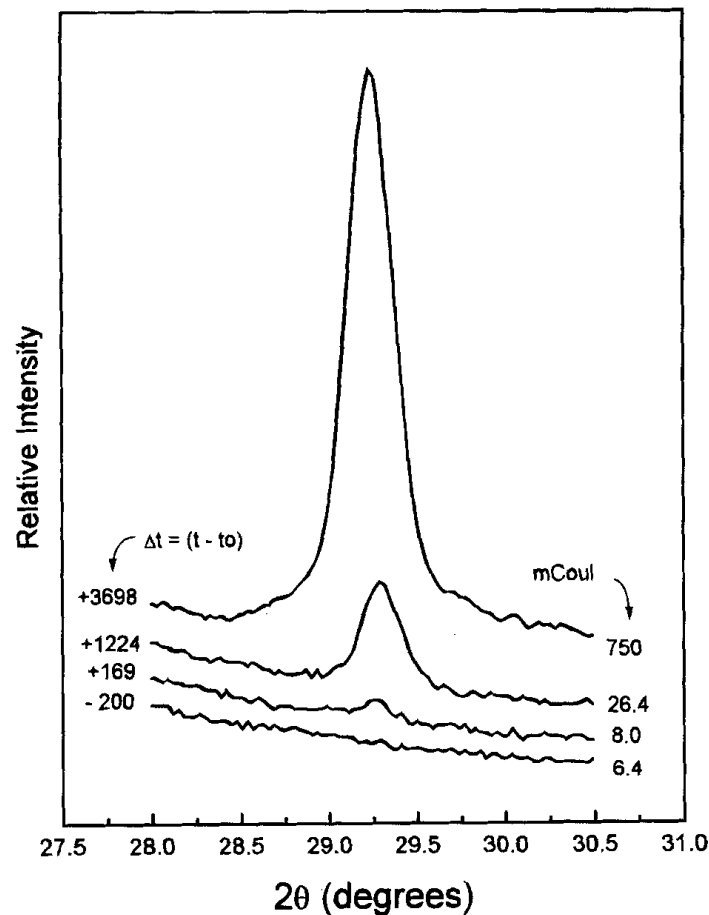


Fig. 10. Low incident angle x-ray diffraction for Ti_2O_3 deposited at an overpotential of 149 mV onto glassy carbon for various deposition times. Crystalline material was observed by diffraction from the (222) planes of Ti_2O_3 after the induction time. Δt represents the deposition time minus the induction time for an overpotential of 149 mV. Positive values of Δt are after the induction time and negative values are before the induction time. The accumulated charge for each trial is shown in millicoulombs on an electrode geometric area of 0.8 cm^2 .

Application of Diffraction Data

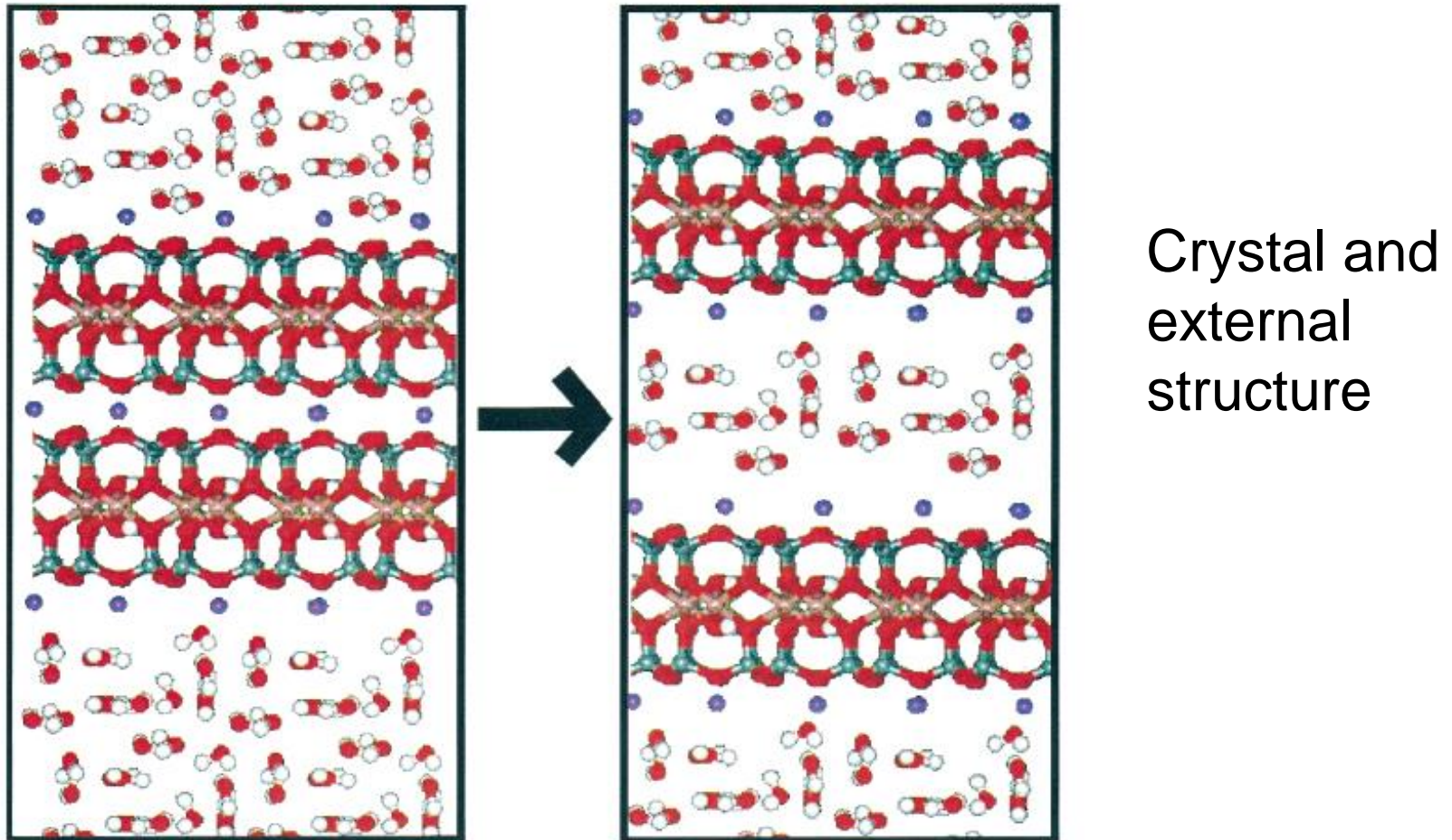


Figure 2. Intercalation scheme of nanoclay mechanics occurring when dissolved platelets (far left) expand their gallery spacing to accommodate intercalants (right). Neutral or cationic species may be intercalated into nanoclay platelets.

Application of Diffraction Data

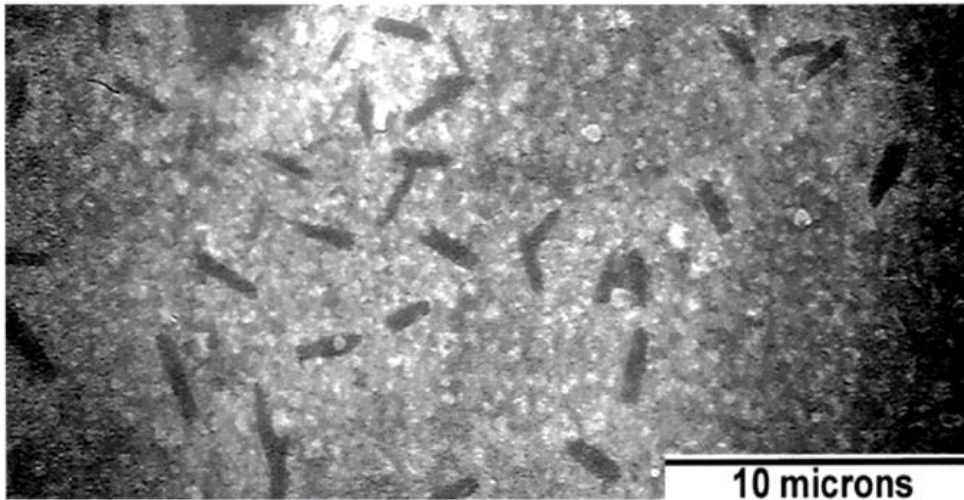


Figure 4. Scanning electron micrograph of a film deposited with a standard electrochemical cell setup at higher stir rates. Montmorillonite layered silicate platelets (dark, rodlike objects) can be seen extruding from the surface of the film.

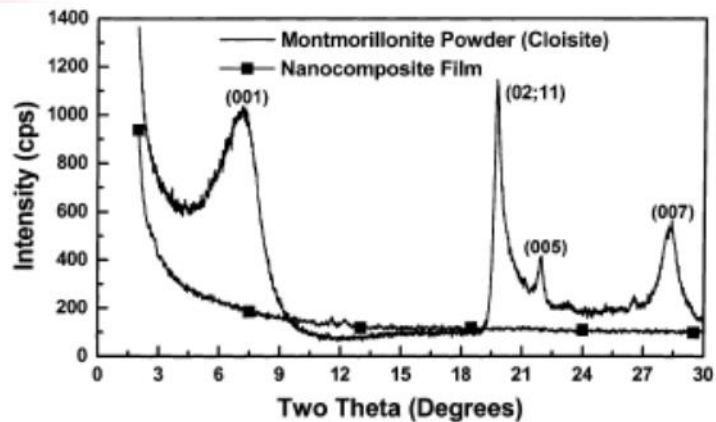
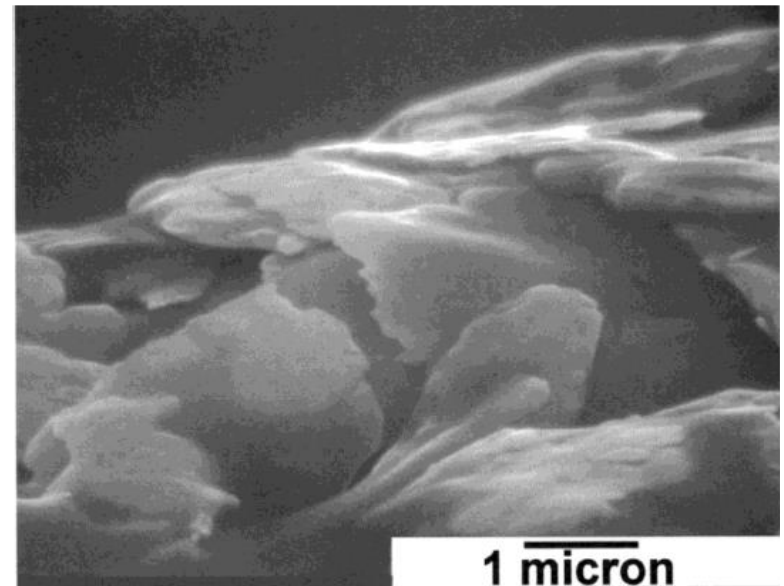


Figure 5. This XRD pattern compares the Ni-MLS film (squares) grown in the third electrochemical cell setup to the pure nanoclay powder (solid line). In the Ni-MLS pattern, the 001 nanoclay peak has disappeared, indicating that the incorporated nanoclay is exfoliated and exists as individual platelets.



Application of Diffraction Data

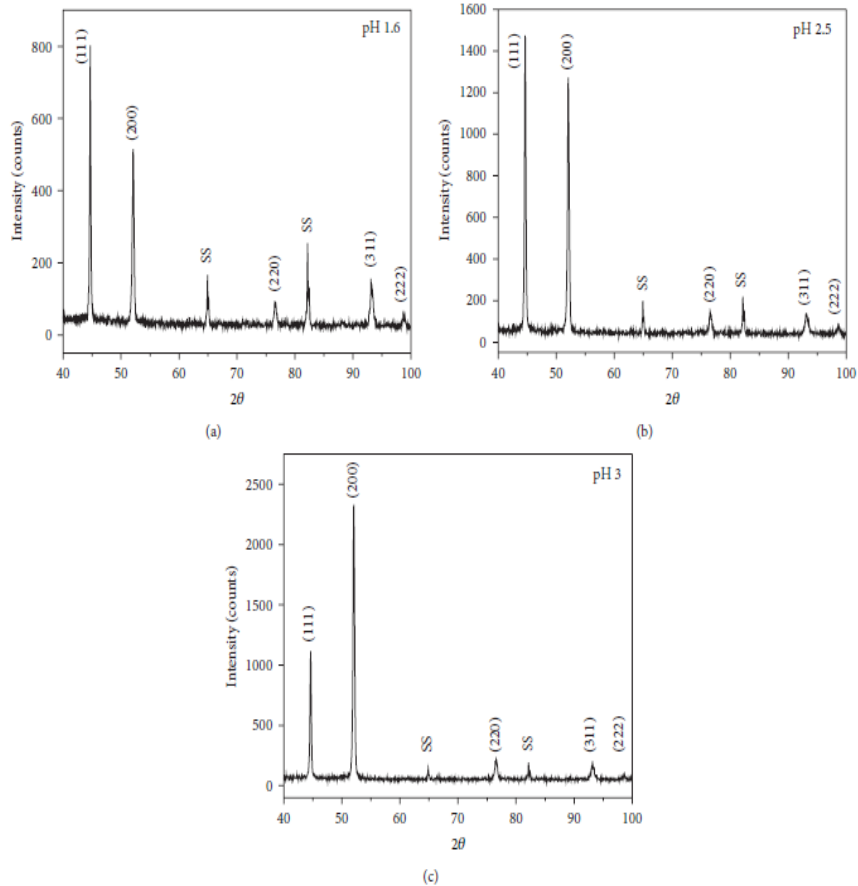


FIGURE 4: X-ray diffraction (XRD) patterns of Ni-MMT (0.5%) films electrodeposited at various pHs (a) 1.6, (b) 2.5 and (c) 3.0 (SS: substrate stainless steel peaks).

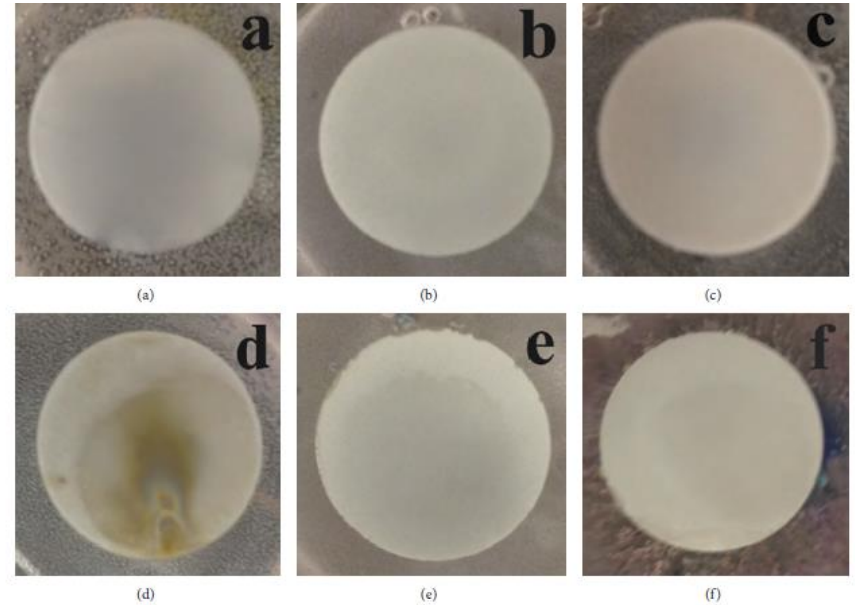


FIGURE 7: Sample images of Ni-MMT coatings electrodeposited from plating baths at various pHs of 1.6, 2.5, and 3.0 before immersion in 3.5% NaCl (a)-(c), and after (d)-(f), respectively.

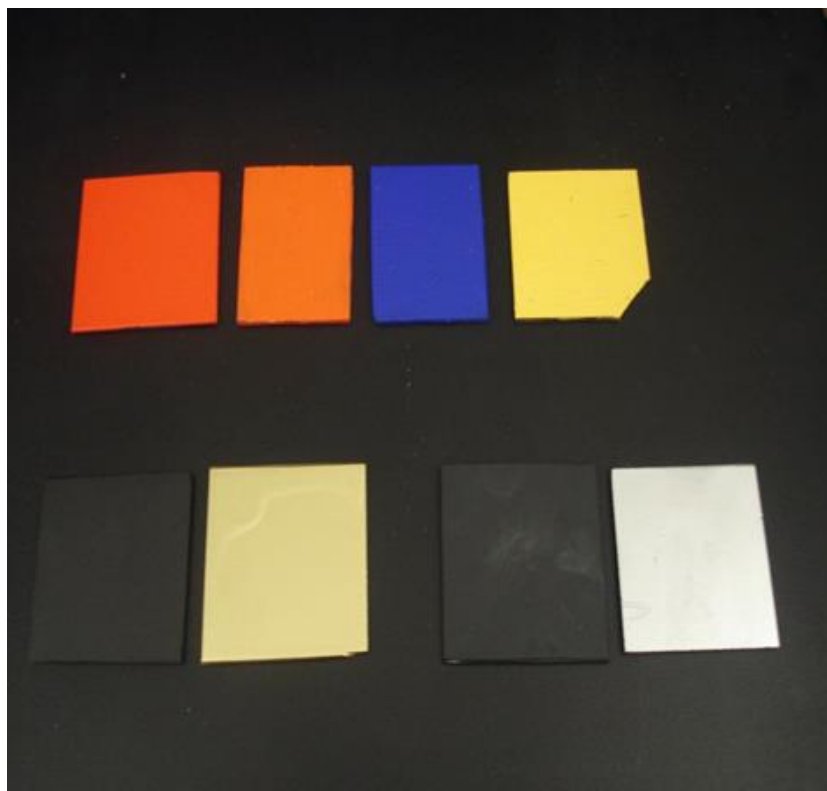
Corrosion Research
Nickel-Clay

Application of Diffraction Data

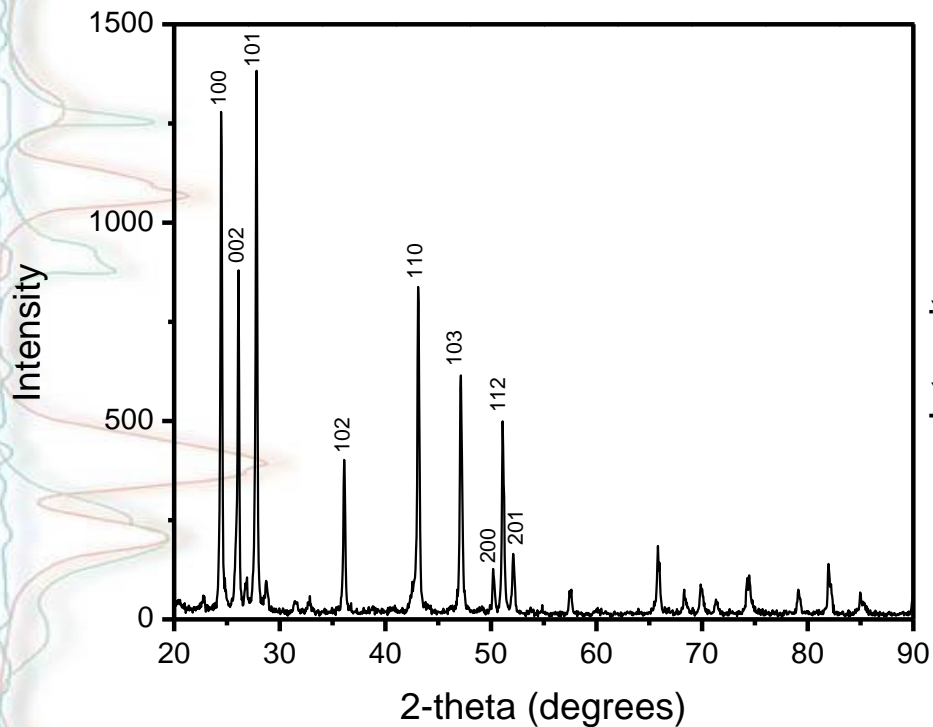
Paint Sample XRD Parameters

| Sample Color Type | Scan Range (degrees) |
|----------------------------|----------------------|
| Cadmium Red Acrylic | 20-90 |
| Scarlet Red Acrylic | 20-60 |
| Cobalt Blue Acrylic | 20-100 |
| Naples Yellow Acrylic | 20-75 |
| Black Matte Spray Paint | 5-35 |
| Metallic Gold Spray Paint | 25-80 |
| Black Matte Auto Paint | 5-35 |
| Metallic Silver Auto Paint | 35-50 |

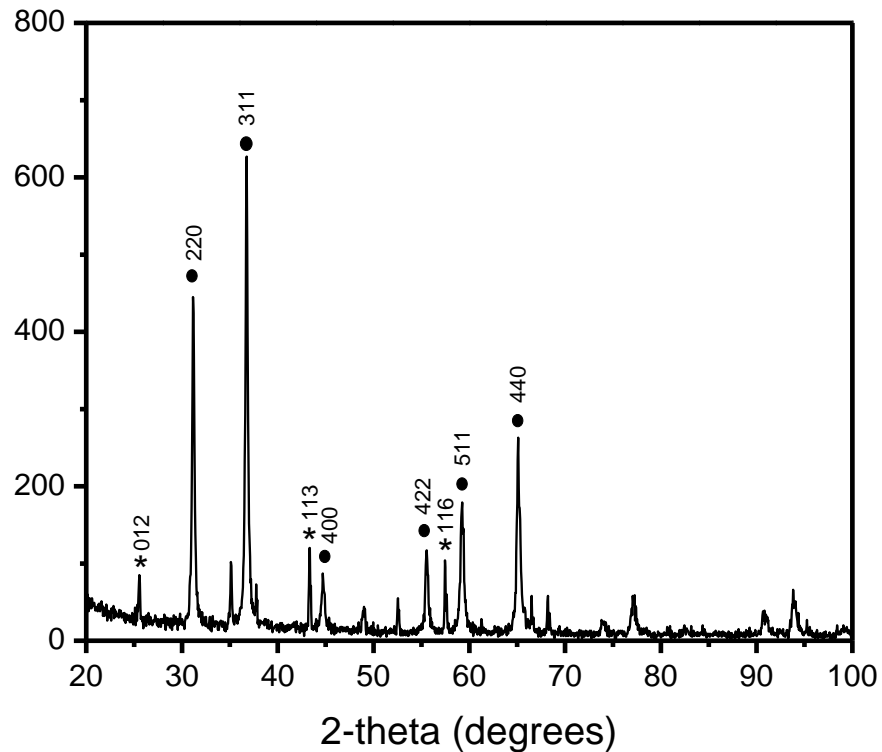
Industry Paints, Epoxies, Polymers



Application of Diffraction Data



cadmium red acrylic paint:
cadmium selenide sulfide



cobalt blue acrylic paint: $*Al_2O_3$, $\bullet Co_3O_4$

Assignments

Read Chapters 1&2&3 from the following textbooks:

- X-ray Diffraction, A Practical Approach by Norton**
- Elements of X-ray Diffraction by Cullity and Stock**
- Introduction to X-ray powder Diffractometry
by Jenkins and Synder**

Read Chapter 9 from:

- Introduction to X-ray powder
Diffractometry by Jenkins and Synder**

