X-ray Diffraction
X-ray Generation

- X-ray tube (sealed)
- Pure metal target (Cu)
- Electrons remover inner-shell electrons from target.
- Other electrons “fall” into hole.
X-ray Generation

- The incoming electron must have enough energy to remove inner 1s electrons from the copper atoms.
- This energy corresponds to the Cu absorption edge.
- The 2s and 2p electrons fall back into the 1s shell and emit the $K\alpha_1$ $K\alpha_2$ lines.
X-ray Spectrum from Tube
Energy Calculations

- Planck’s constant \( h = 6.6 \times 10^{-34} \) joule-sec
- 1 electron-volt = 1.6016 \times 10^{-19} \) joule
- Speed of light \( c = 3.0 \times 10^8 \) m/s
- Photon frequency \( \nu = c/\lambda \)
- Photon Energy \( E = h\nu = hc/\lambda \)
Energy Calculations

- What is the minimum potential in KV that is required to excite Cu K-series radiation from a Cu-target X-ray tube?

- Absorption edge of Cu = 1.380Å

- \[ E = \frac{hc}{\lambda} = \frac{(6.60 \times 10^{-34})(3\times10^8)}{(1.380\times10^{-10})} \]

- \[ E = 1.435\times10^{-15} \text{ joule} \]

- \[ E = 1.435\times10^{-15} / 1.6016\times10^{-19} = 8958 \text{ ev} \]

- The potential on the tube must exceed 8.958 KV
Diffraction

- Diffraction is the coherent scattering of waves from a periodic array of scatterers.
- The wavelength of light is about half a micron.
- Light is diffracted by the tracks in a CD.
- The wavelengths of X-rays is about the same as the interatomic distances in crystals.
- Crystals diffract X-rays.
X-Ray Diffraction

- Atoms separated by distance $d$ will scatter in phase when the path length difference is an integral number of wavelengths.

- Path length difference $B-C \ D = n\lambda$

- $n\lambda = 2d \sin \theta$
X-ray Diffraction Experiment

- We use the ‘monochromatic’ Kα₁-2 lines for our diffraction experiment.
- The wavelength is 1.5405 Å
- We use a diffracted beam monochromator to clean up the X-rays entering the detector.
- We use a powdered sample so that all orientations are present in the sample.
- We move the detector through angle 2θ.
Miller Indices

- The real use of Miller indices is to describe diffraction planes.
- For a lattice plane with Miller indices $h \ k \ l$ in an orthorhombic lattice $a \ b \ c$,
  - $d = 1 / [(h/a)^2+(k/b)^2+(l/c)^2]^{1/2}$
- For cubic:
  - $d = a/[h^2+k^2+l^2]^{1/2}$
**Diffraction Calculations**

- For forsterite \( a = 4.75; \ b = 10.20; \ c = 5.98\text{Å} \)
- Calculate \( 2\theta \) for the (201) lattice spacing for CuK\(\alpha \) (\( \lambda = 1.5405\text{Å} \))
- \( d = \frac{1}{\left[ \left( \frac{h}{a} \right)^2 + \left( \frac{k}{b} \right)^2 + \left( \frac{l}{c} \right)^2 \right]^{1/2}} \)
- \( d = \frac{1}{\left[ \left( \frac{2}{4.75} \right)^2 + \left( \frac{1}{5.98} \right)^2 \right]^{1/2}} \)
- \( d = \frac{1}{0.4530} = 2.207\text{Å} \)
- \( 2\theta = 2 \sin^{-1} \frac{\lambda}{2d} = 2^* \sin^{-1} \left( \frac{1.5405}{4.414} \right) \)
- \( 2\theta = 2 \times 20.43 = 40.86^\circ \)
XPOW

- XPOW uses the unit cell and atom position data to calculate the diffraction pattern.
- Intensities can be calculated knowing the position and scattering characteristics of each atom.
- \( F_{hkl} = \) square root of integrated intensity.
- \( f_j = \) scattering of atom \( j \) at angle \( 2\theta \)
- Atom \( j \) located at fractional coordinates \( x_j, y_j, z_j \).

\[
F_{hkl} = \sum_{j=1}^{n} f_j e^{2\pi i (hx_j + ky_j + lz_j)}
\]
Uses of X-ray Powder Diffraction

- Mineral identification
- Determination of Unit Cell Parameters
- Modal (phase percentage) Analysis
- Crystal Structure Determination