X-ray Diffraction and Crystal Structures

Week of October 18, 2010

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(Physics 4780)

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X-Ray Generation

X-rays are electromagnetic radiation with wavelength $\sim 1 \, \text{Å} = 10^{-10} \, \text{m}$ (visible light $\sim 5.5 \times 10^{-7} \, \text{m}$)

X-ray generation

X-ray wavelengths too short to be resolved by a standard optical grating

$$\theta = \sin^{-1} \frac{m\lambda}{d} = \sin^{-1} \frac{1 \times (0.1 \, \text{nm})}{3000 \, \text{nm}} = 0.0019^\circ$$
The most common metal used is copper, which can be kept cool easily, due to its high thermal conductivity, and which produces strong $K_\alpha$ and $K_\beta$ lines. The $K_\beta$ line is sometimes suppressed with a thin (~10 µm) nickel foil.

- **K-alpha ($K_\alpha$)** emission lines result when an electron transitions to the innermost "K" shell (principal quantum number 1) from a 2p orbital of the second or "L" shell (with principal quantum number 2).

- The $K_\alpha$ line is actually a doublet, with slightly different energies depending on spin-orbit interaction energy between the electron spin and the orbital momentum of the 2p orbital.

\[
\lambda(K_\alpha) = 0.154 \text{ nm} \\
\lambda(K_\beta) = 0.139 \text{ nm}
\]

$K_{\alpha}$ and $K_{\beta}$ X-ray lines

from Preston and Dietz, p. 191.
X-Ray diffraction

\[ d \sin \theta \]
X-Ray Diffraction -- Bragg’s Law

Diffraction of x-rays by crystal: spacing $d$ of adjacent crystal planes on the order of 0.1 nm

→ three-dimensional diffraction grating with diffraction maxima along angles where reflections from different planes interfere constructively

$$2d \sin \theta = m\lambda \text{ for } m = 0, 1, 2, \ldots$$
Bragg occupied the Cavendish chair of physics at the University of Leeds from 1909. He continued his work on X-rays with much success. He invented the X-ray spectrometer and with his son, William Lawrence Bragg, then a research student at Cambridge, founded the new science of X-ray analysis of crystal structure.

In 1915 father and son were jointly awarded the Nobel Prize in Physics for their studies, using the X-ray spectrometer, of X-ray spectra, X-ray diffraction, and of crystal structure.

Interplanar spacing $d$ is related to the unit cell dimension $a_0$

$$5d = \sqrt{\frac{5}{4}} a_0^2 \quad \text{or} \quad d = \frac{a_0}{20} = 0.2236a_0$$

Not only can crystals be used to separate different x-ray wavelengths, but x-rays in turn can be used to study crystals, for example determine the type of crystal ordering and $a_0$. 

X-Ray Diffraction, cont’d
Crystal structure, lattice planes, and Miller indices

Planes with different Miller indices in cubic crystals. The inverse of these fractional intercepts yields the Miller indices $h, k, l$.
Crystal structure and Miller indices

Planes with different Miller indices in cubic crystals.

from http://en.wikipedia.org/wiki/Miller_index
Crystal structure and Miller indices

http://www.msm.cam.ac.uk/doitpoms/tlplib/miller_indices/lattice_index.php
Rock salt (cubic) crystal structure

$$d_{hkl} = \frac{a_0}{\sqrt{h^2 + k^2 + l^2}}$$

Structure factor for NaCl:

$$F = [f_{Na} + f_{Cl} e^{i\pi(h+k+l)}] \left[1 + e^{i\pi(h+k)} + e^{i\pi(h+l)} + e^{i\pi(k+l)}\right]$$

- $F = 4(f_{Na} + f_{Cl})$ if $h, k, l$ are even
- $F = 4(f_{Na} - f_{Cl})$ if $h, k, l$ are odd
- $F = 0$ if $h, k, l$ are mixed

X-Ray diffraction: a practical approach, by C. Suryanarayana, M. Grant Norton
X-Ray diffraction (XRD) pattern (diffractogram) from NaCl

Miller indices: The peak is due to X-ray diffraction from the \{220\} planes.

Diffraction angle \(2\theta\) (degrees)

\[
d_{hkl} = \frac{a_0}{\sqrt{h^2 + k^2 + l^2}}
\]

http://web.pdx.edu/~pmoeck/phy381/Topic5a-XRD.pdf
LiF diffractogram (Cu K$\alpha$)
TEL-X-Ometer

\[ K_{\alpha 1} \quad 1.540 \, \text{Å} \]
\[ K_{\alpha 2} \quad 1.544 \, \text{Å} \]
\[ K_{\beta} \quad 1.392 \, \text{Å} \]