X-ray diffraction

By- Dr. Ekta Khare
Department of Microbiology,
School of Life sciences and Biotechnology,
CSJM University, Kanpur

https://web.pdx.edu/~pmoeck/phy381/Topic5a-XRD.pdf

https://www.slideshare.net/slideshow/xray-diffraction-instrumentation/75020328

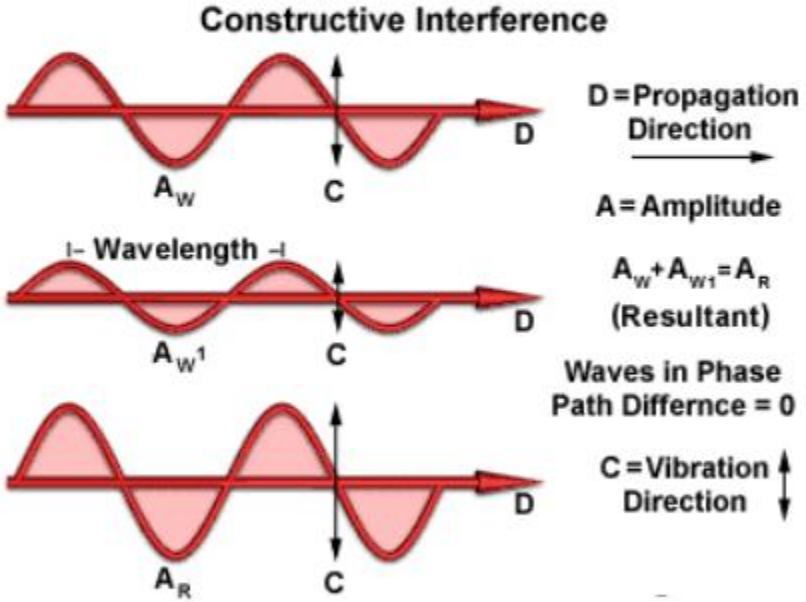
XRD

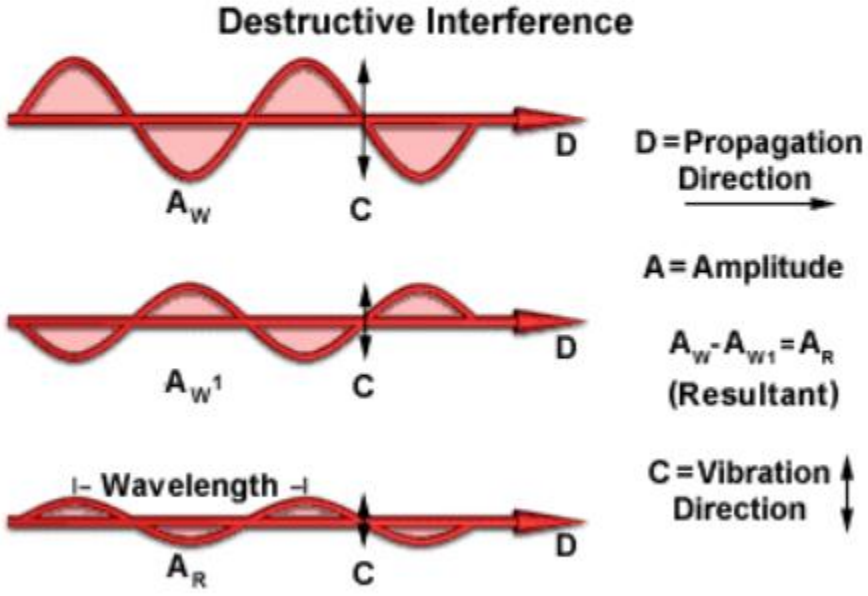
- English physicists Sir W.H. Bragg and his son Sir W.L. Bragg developed a relationship in 1913 to explain why the cleavage faces of crystals appear to reflect X-ray beams at certain angles of incidence (theta, θ).
- The variable d is the distance between atomic layers in a crystal, and the variable lambda λ is the wavelength of the incident X-ray beam; n is an integer.
- This observation is an example of X-ray wave interference, commonly known as X-ray diffraction (XRD), and was direct evidence for the periodic atomic structure of crystals postulated for several centuries.
- The Braggs were awarded the Nobel Prize in physics in 1915 for their work in determining crystal structures beginning with NaCl, ZnS and diamond.

Bragg's Law

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

Constructive and Destructive Interference of Waves



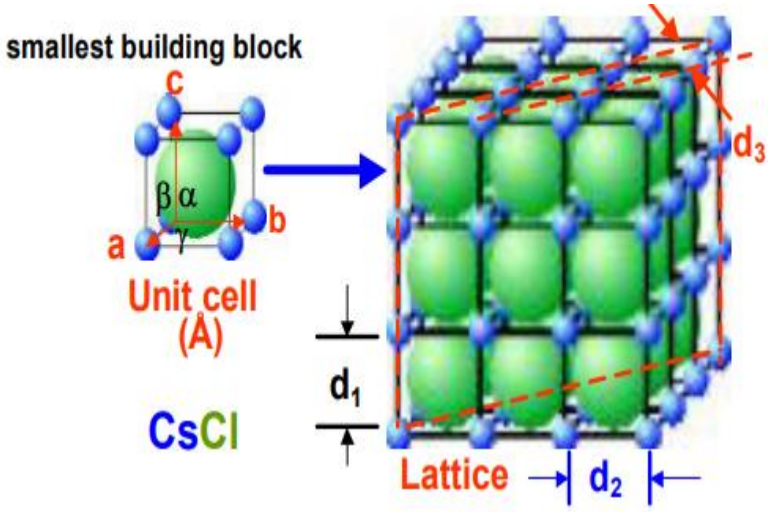


Constructive Interference
In Phase

Destructive Interference
Out of Phase

Why XRD?

- Measure the average spacings between layers or rows of atoms.
- Determine the orientation of a single crystal or grain.
- Find the crystal structure of an unknown material .
- Measure the size, shape and internal stress of small crystalline regions.



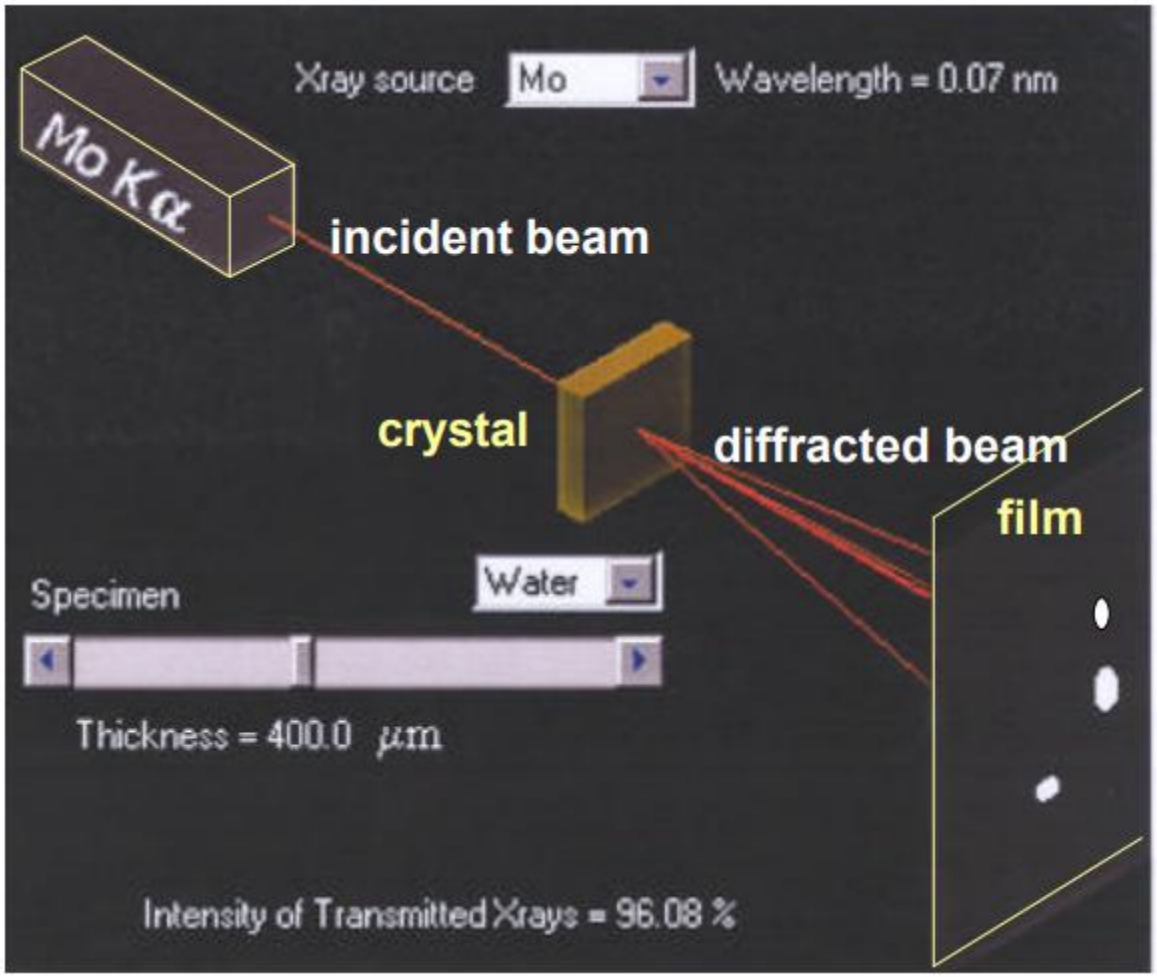
A crystal consists of a periodic arrangement of the unit cell into a lattice. The unit cell can contain a single atom or atoms in a fixed arrangement.

Crystals consist of planes of atoms that are spaced a distance **d** apart, but can be resolved into many atomic planes, each with a different **d**-spacing.

a,b and c (length) and α, β and γ angles between a,b and c are lattice constants or parameters which can be determined by XRD.

X-ray Diffraction (XRD)

- The atomic planes of a crystal cause an incident beam of X-rays to interfere with one another as they leave the crystal.
- The phenomenon is called X-ray diffraction.



Instrumentation

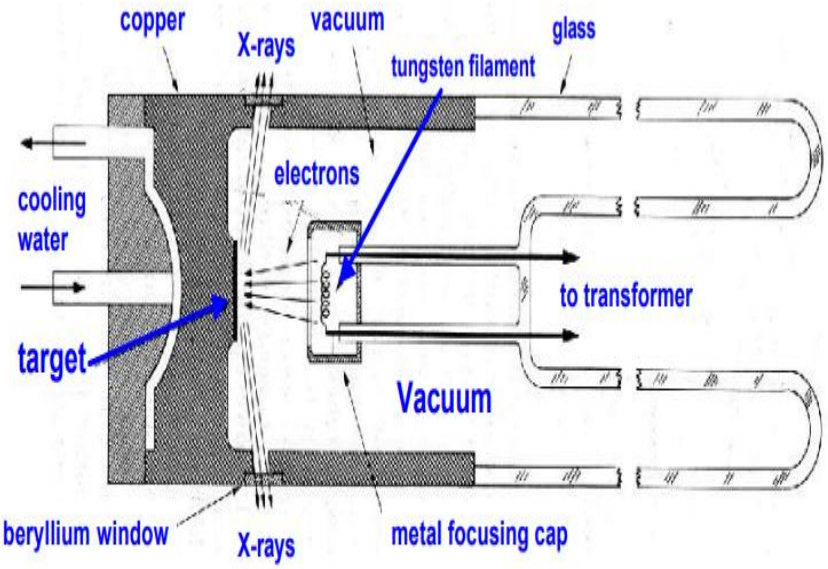
- Radiation source
- Collimator
- Monochromator
- Sample handling
- Detector
- X ray diffraction device

X-ray Tube: radiation source

Features & Functioning of XRT

- Composed of evacuated tube possessing cathode (tungsten filament) at one end & anode(metal target) at another end.
- Passage of current through tube causes tungsten filament to glow & emits electron.
- Among the two electrodes large voltage difference is applied, causing electrons to move at high velocity from filament and strike to anode.
- Due to high velocity impact of electrons on to the target, inner shell electrons of metal gets dislodge, which causes the outer shell electrons to jump to a lower energy shell to replace the dislodge electrons.
- These electronic transitions results in the generation of X-rays. The produced X-rays are allowed to move through a window of X-ray tube.

Cross section of sealed-off filament X-ray tube



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.

Collimator

- A device that is used for narrowing of a beam of particles or waves.
- Collimator makes, random directional X-rays to be narrow and parallel.

Monochromators

A device that is used for removal of unwanted radiation.

1. Filters Monochromators
2. Crystal Monochromators

Detectors

X-rays can be detected using two types of detectors:

1. Photographic Detectors
2. Counter Detectors
   a) Geiger Muller Tube Counter
   b) Proportional Counter
   c) Scintillator Counter
   d) Semiconductor Detector