X-Ray Diffraction and Bragg's Law

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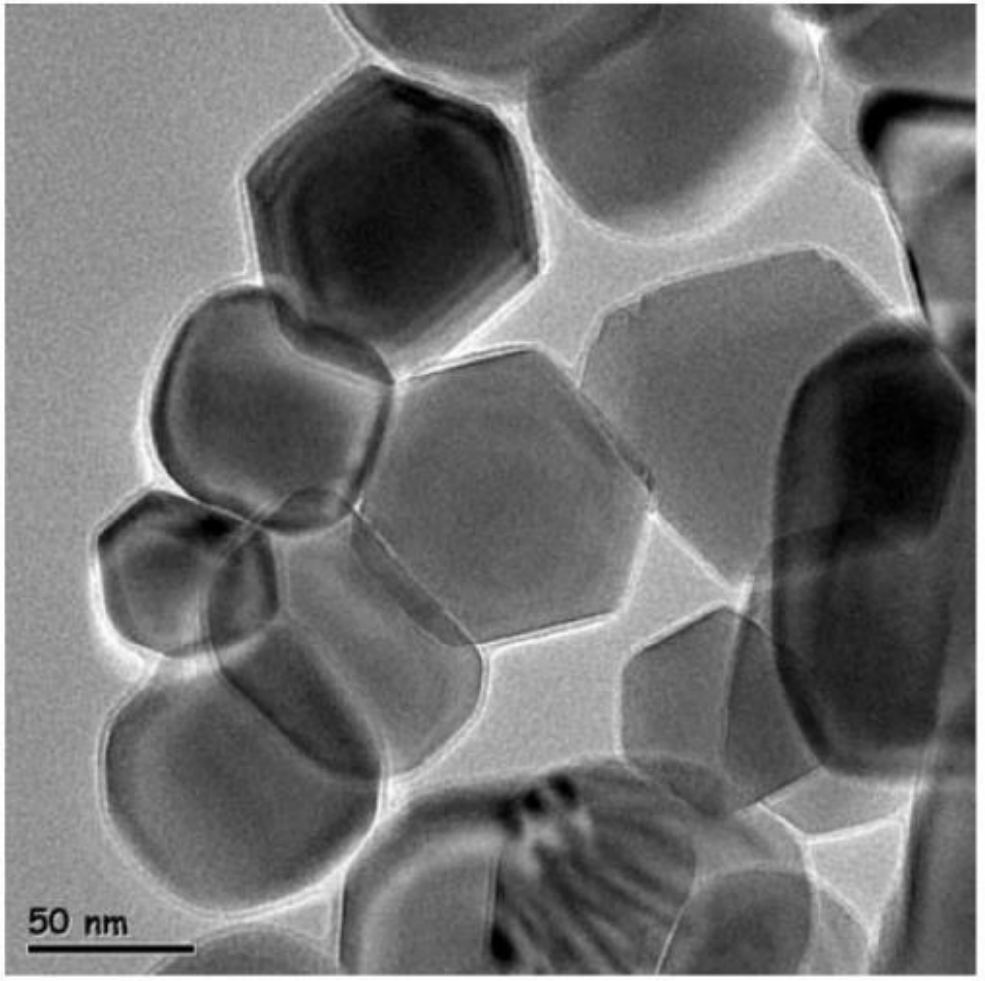
Structural Analysis of Solid

- Structure of solids is difficult to analyze by direct measurements.
- Electron microscope with a high resolution can't still show clear image in atomic level.
- It is convenient to analyze crystal structure by observing the optical phenomena such as reflection, scattering, diffraction, etc. by the help of either high energy particles or photons.

TEM image of ZnO nanoparticles



Lee WH et al., "Round-robin test on thermal conductivity measurement of ZnO nanofluids and comparison of experimental results with theoretical bounds", Nanoscale Res Lett (2011)

Crystal Structure Studies

1. X-rays; photon energy ~ 50 keV, very short wavelength and high penetration power.
2. Neutrons, energy ~ 0.08 eV, usually preferable for magnetic crystals, because in the case of non-magnetic materials it interacts only with the nuclei of the constituent atoms
3. Electrons, energy ~ 100 eV, penetrates relatively short distance in the crystal

Properties of X-rays

- X-rays travel in straight lines.
- They cannot be deflected by electric or magnetic field.
- They have high penetration power due to short wavelength ranging from 0.1 to 100 Å.
- They exhibit almost all optical phenomena like reflection, refraction, diffraction, etc.
- They show effect in photographic plates (films).
- Fluorescent materials glow when X-rays are directed at them.
- Ionization of gas results when X-ray beam is passed through it.

Advantages of X-ray Diffraction Technique

- It is very cost effective and convenient.
- It is very user friendly.
- In this technique no vacuum is required.
- X-rays are not much absorbed in air.

Disadvantages of X-ray Diffraction Technique

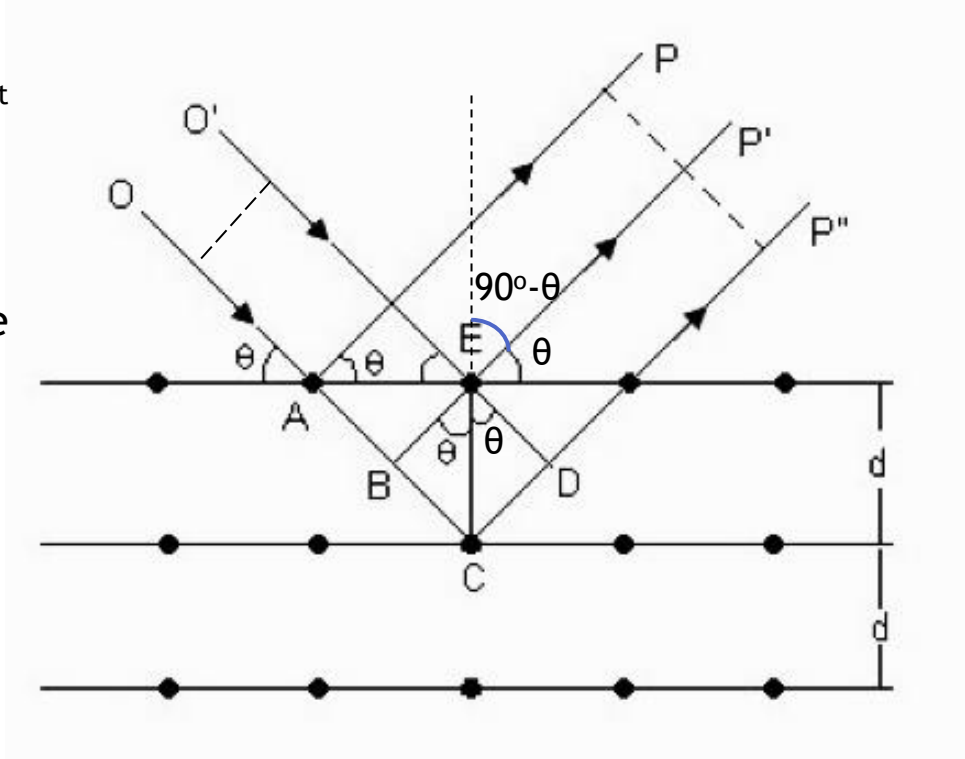
- X-rays do not interact very strongly with lighter elements.

X-ray diffraction and Bragg's law

- OA and O'E: Incident rays
- AP and EP': Reflected from the 1st surface
- CP": Reflected from the 2nd surface
- θ: Angle at which OA and O'E are incident on the 1st surface
- EC=d= interplanar spacing
- O'EP' and OAP are the same.
- OCP" is longer than the path O'EP' by an amount, Δ= BCD = BC + CD
- Now, from the right angle triangles EBC and EDC, we have BC=dsinθ=CD; So, Δ = 2d sinθ
- If two consecutive planes scattered in phase

  i.e. Δ = nλ

  where λ= wavelength of X-rays and n = 0, 1, 2 ... =order of reflection



Bragg's law: nλ= 2d sinθ

W.L. Bragg in 1913.

Bragg's condition

$(\sin\theta)_{max}=1,\ \frac{n\lambda}{2d} \leq 1$

Experimental X-ray diffraction Methods

To satisfy Bragg's law, it is necessary to vary either the angle of inclination of the specimen to the beam or the wavelength of the radiation. The three standard methods of X-ray crystallography are-

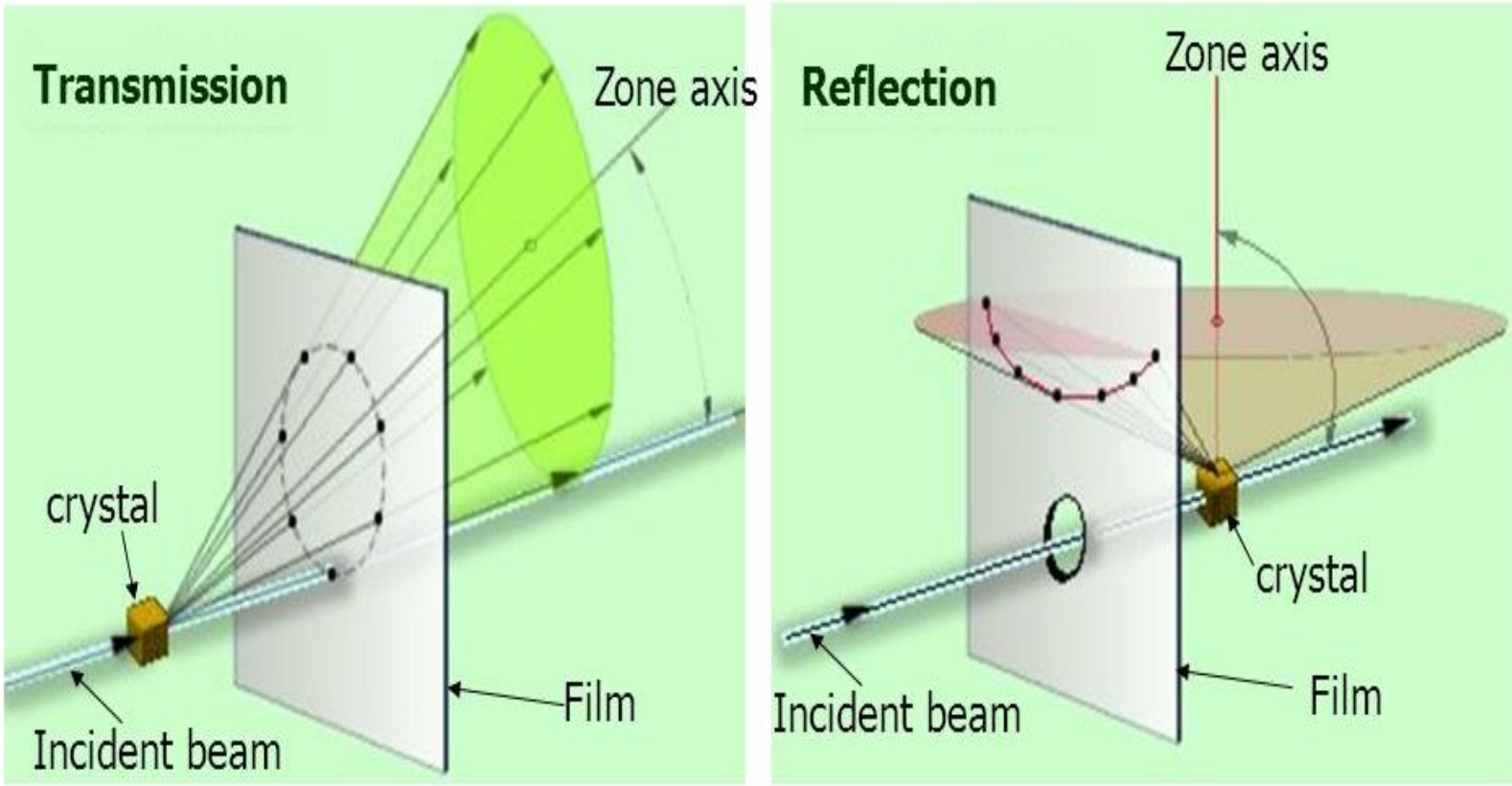
a) **Laue Method**: A stationary single crystal is irradiated by a range of X-ray wavelengths.

b) **Rotating crystal Method**: A single crystal specimen is rotated in a beam of monochromatic X-rays.

c) **Powder Method**: A polycrystalline powder specimen is kept stationary in a beam of monochromatic radiation.

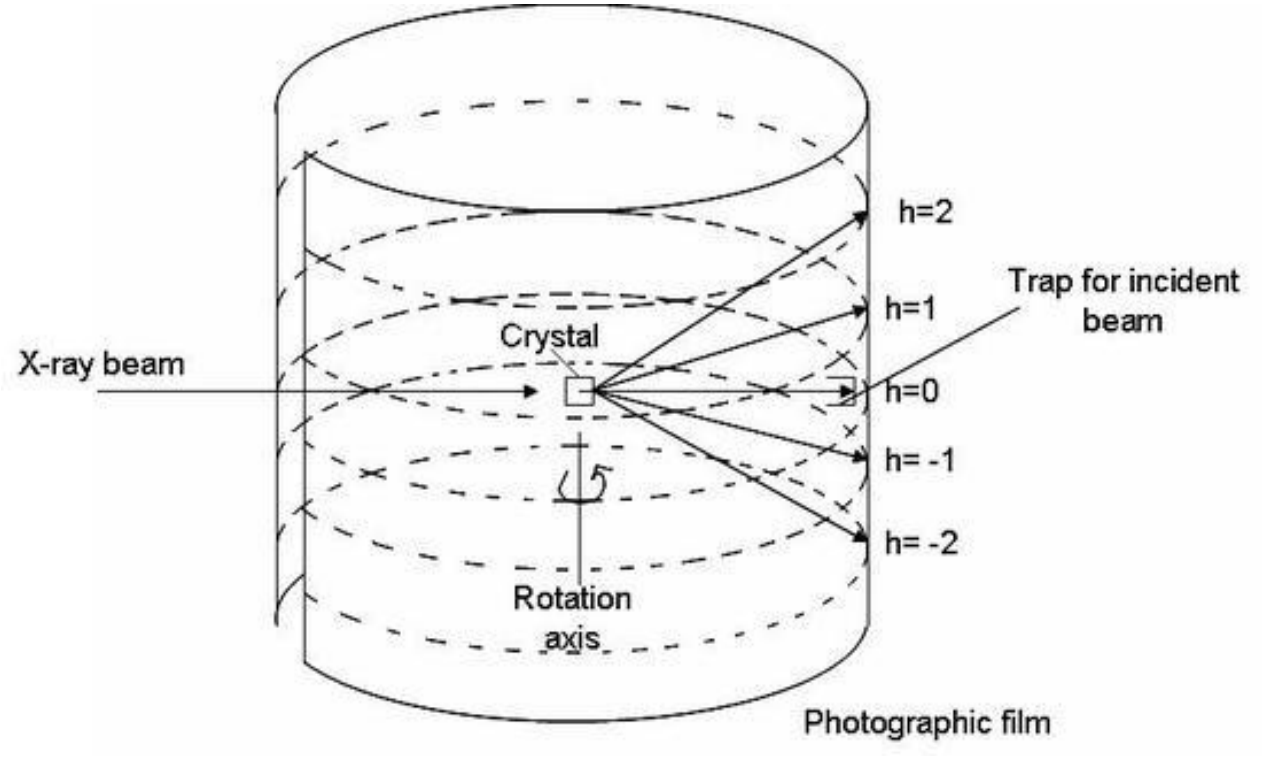
Of these techniques, Laue method is used only for known crystal orientation measurement.

Laue Method

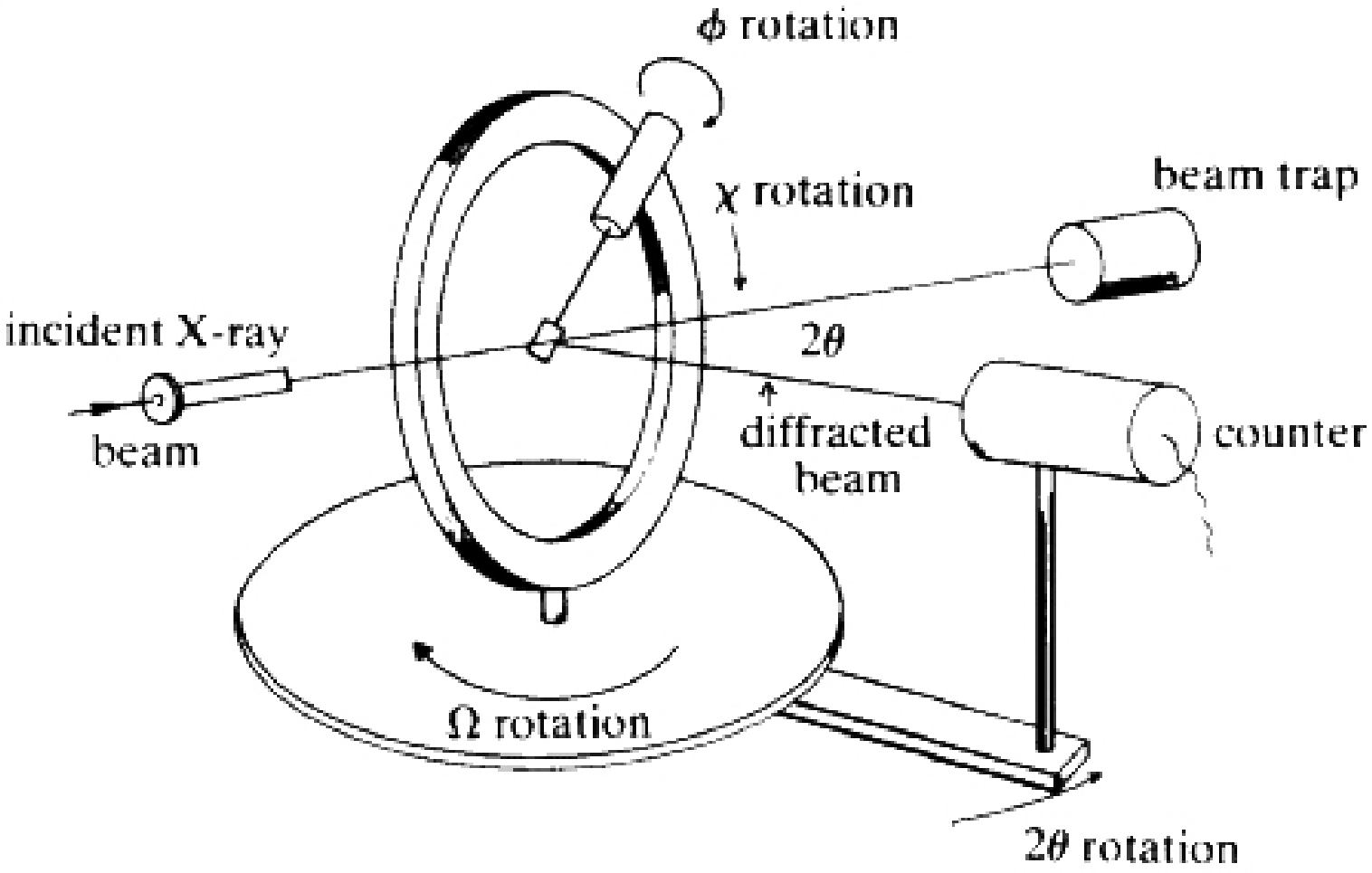


- Uses Single crystal
- Uses White Radiation
- Used for determining crystal orientation and quality

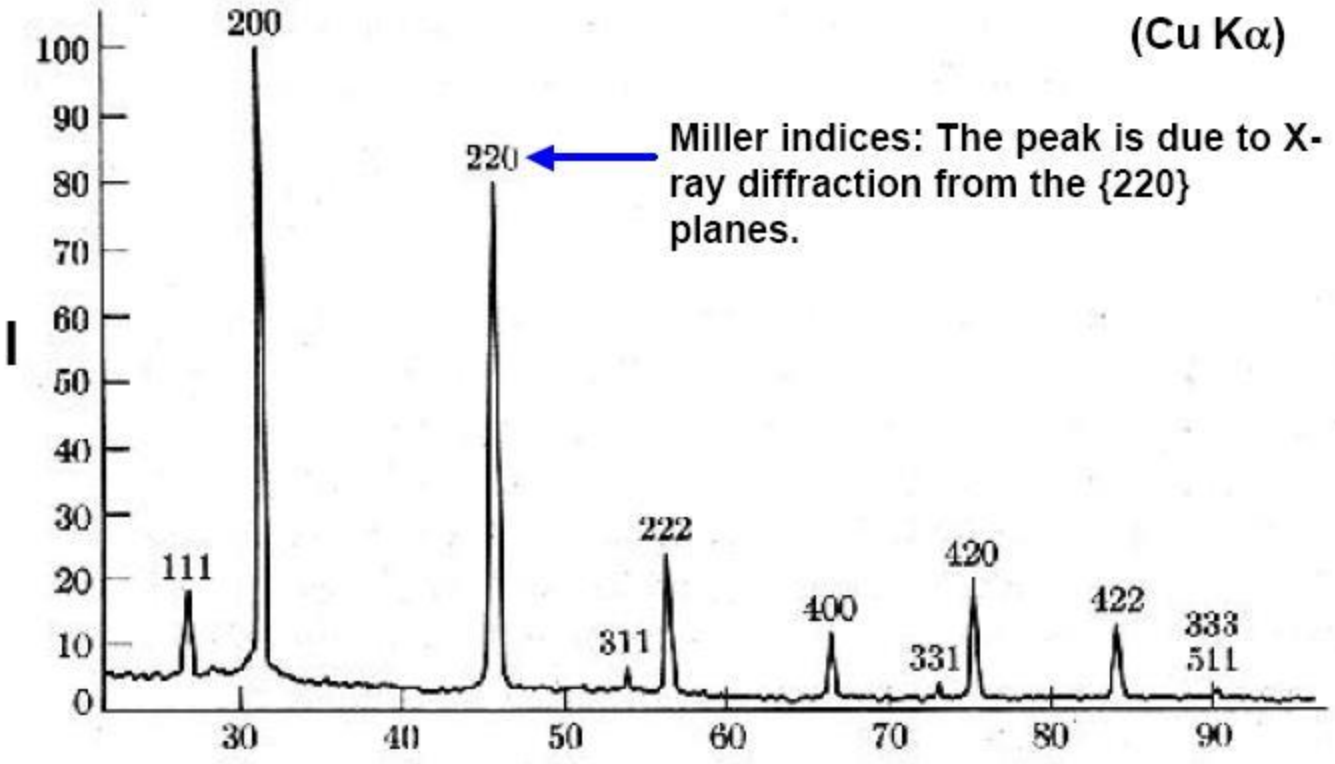
Rotating Crystal Method

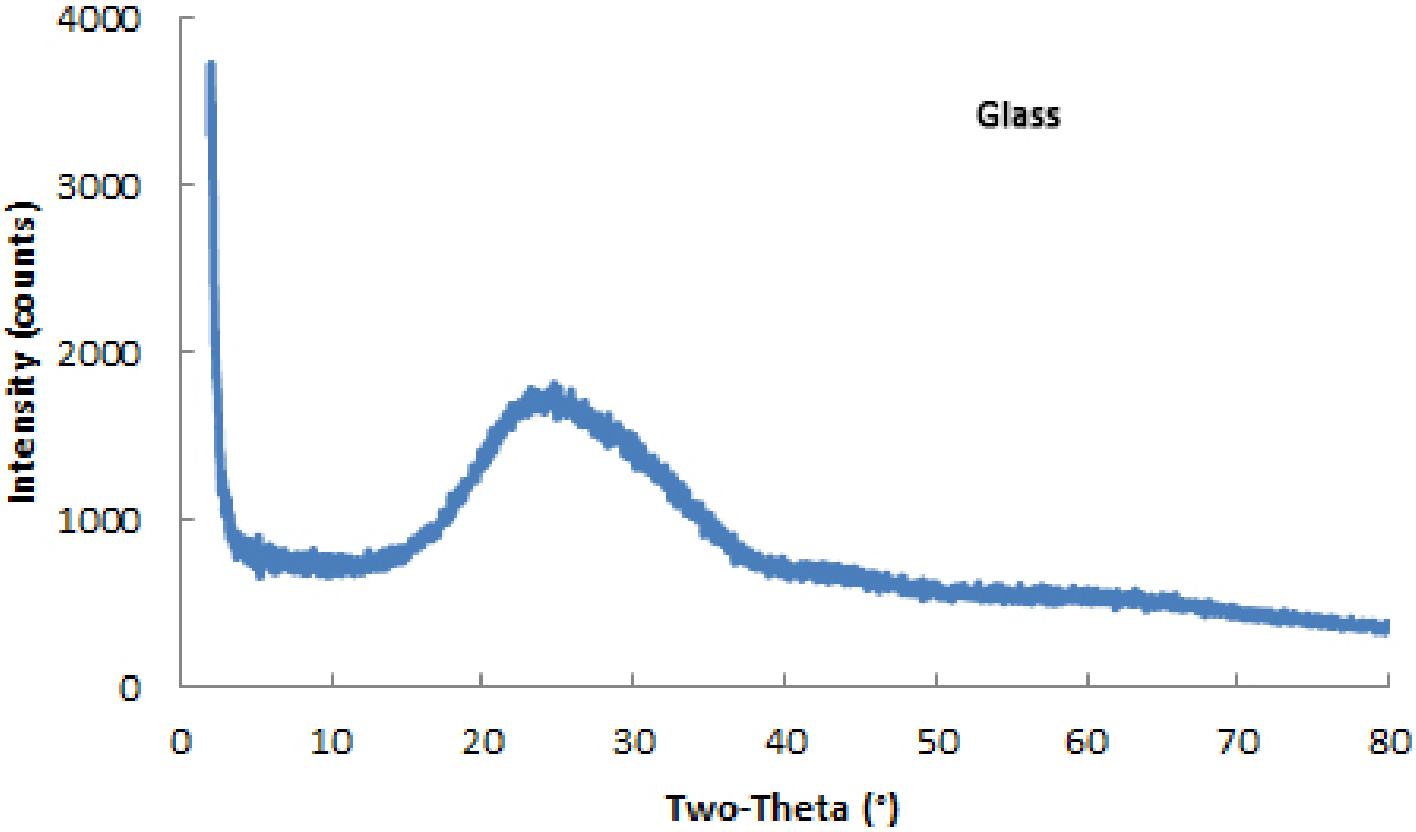


Powder Method



XRD Pattern of NaCl Powder




Glass
Intensity (counts)
4000
3000
2000
1000
0
0
10
20
30
40
50
60
70
80
Two-Theta (°)