Basics of X-ray Diffraction Technique

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Introduction

Max Von Laue

- X-rays were discovered by Wilhelm Roentgen (aka Roentgen rays) in 1895
- X-ray diffraction in crystals was discovered by Max Von Laue in 1912

- X-ray wavelength range: 0.01-10 nm
- Penetrating power depends on energy

Two types of X-rays

- Hard X-rays: high frequency & energy
- Soft X-rays: lower frequency & energy

Introduction

X-rays: short wavelength electromagnetic radiations produced by deceleration of high energy electrons or by electronic transitions of electrons in the inner orbital of atoms

Principle: X-ray diffraction is based on the **constructive interference** of monochromatic X-rays and crystalline materials (samples)

- X-rays generated by cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate and directed towards sample
- Interaction of incident rays with sample produces **constructive interferences** when conditions satisfy Bragg's Law

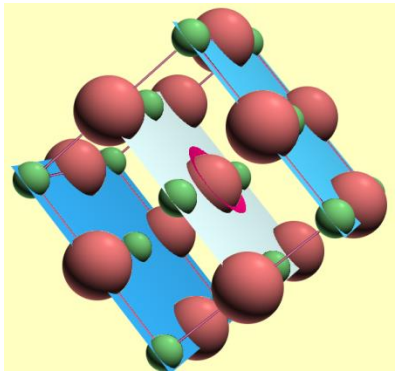
X-ray diffraction (XRD)

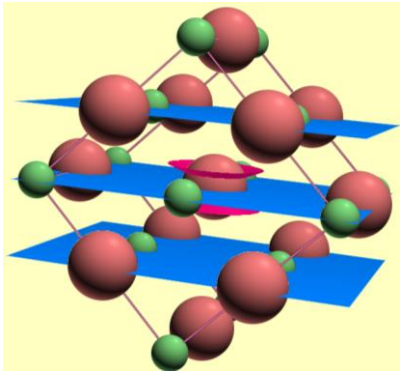
- Non-destructive
- Analysing wide range of materials
  - Fluids
  - Metals
  - Minerals
  - Polymers
  - Plastics
  - Pharmaceuticals
  - Thin-film coating
  - Ceramics
  - Solar cells
  - Semiconductors

Crystal structure

- Crystalline materials are characterized by orderly periodic arrangements of atoms which form a crystal lattice that extends in all directions

The (200) planes of atoms in NaCl

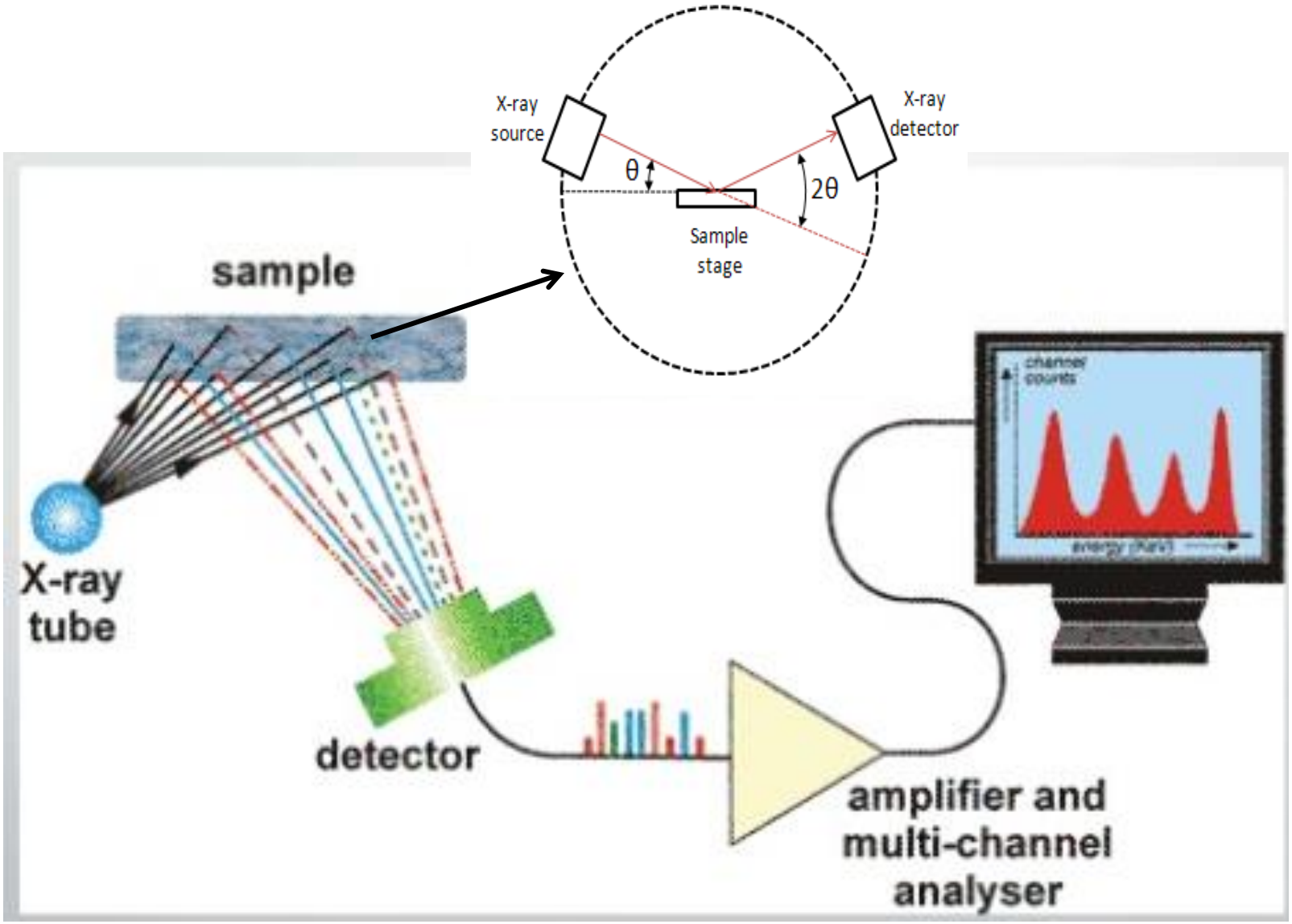




The (220) planes of atoms in NaCl

- The unit cell is the basic repeating unit that defines a crystal.
- Parallel planes of atoms intersecting the unit cell are used to define directions and distances in the crystal.
  - These crystallographic planes are identified by Miller indices.

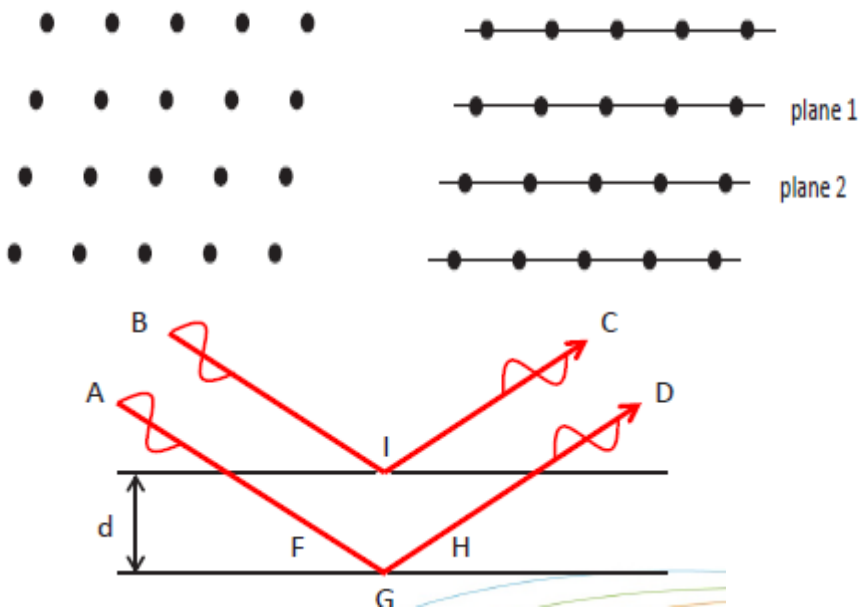
Instrumentation



Diffraction on crystals

- **Bragg condition**

Diffraction by 3D lattice of points is equivalent to a reflection of the incident beam on a family of net planes



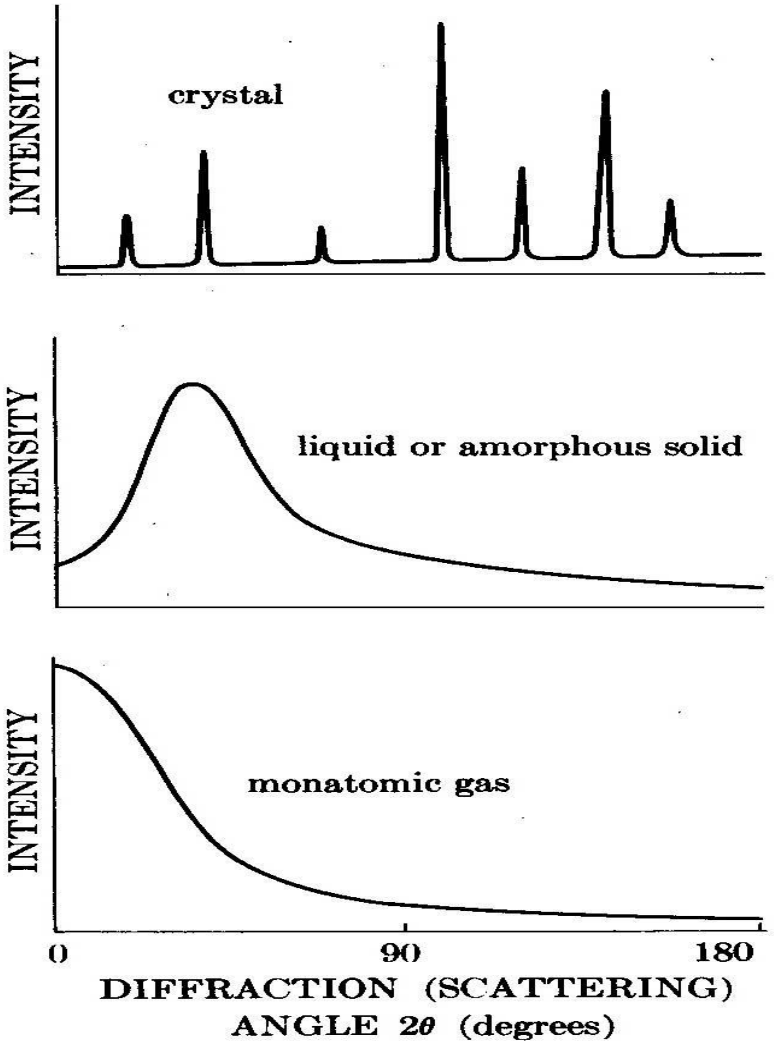
The incident beam IC & GD scattered by 2 adjacent planes must be in phase to get constructive interference

FG + GH = nλ
With FG = GH = d sinθ

nλ = 2d sinθ

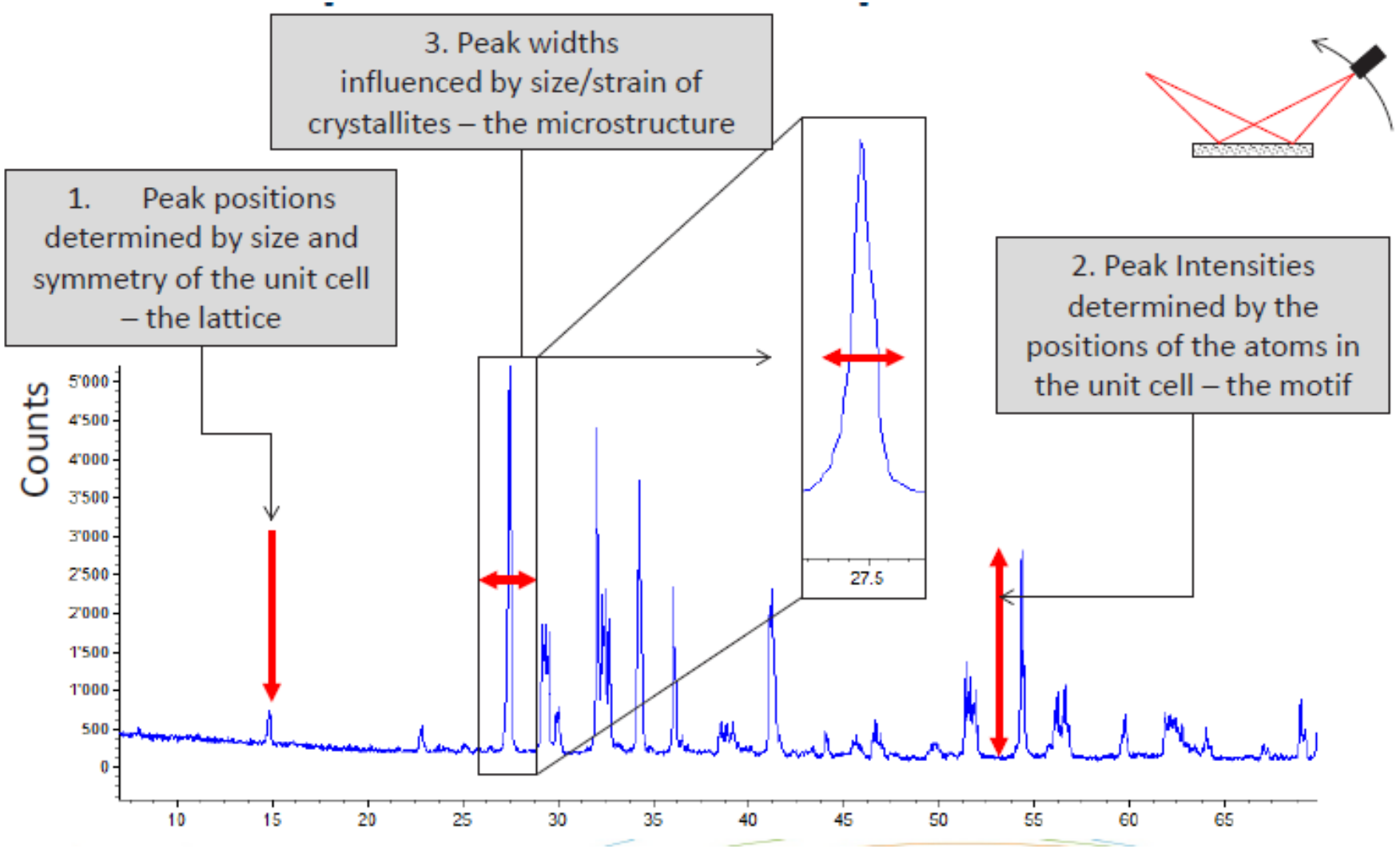
Bragg's Law

Diffractograms of different materials



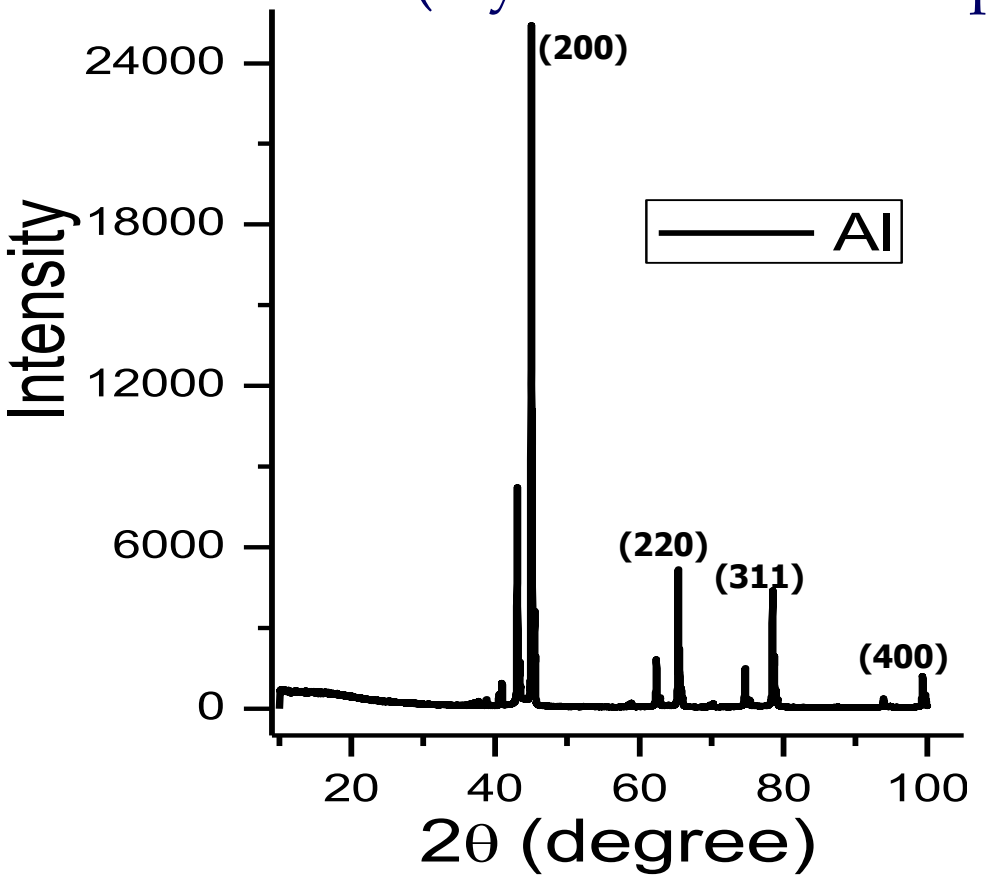
(From "Elements of X-ray Diffraction", B.D. Cullity, Addison Wesley)

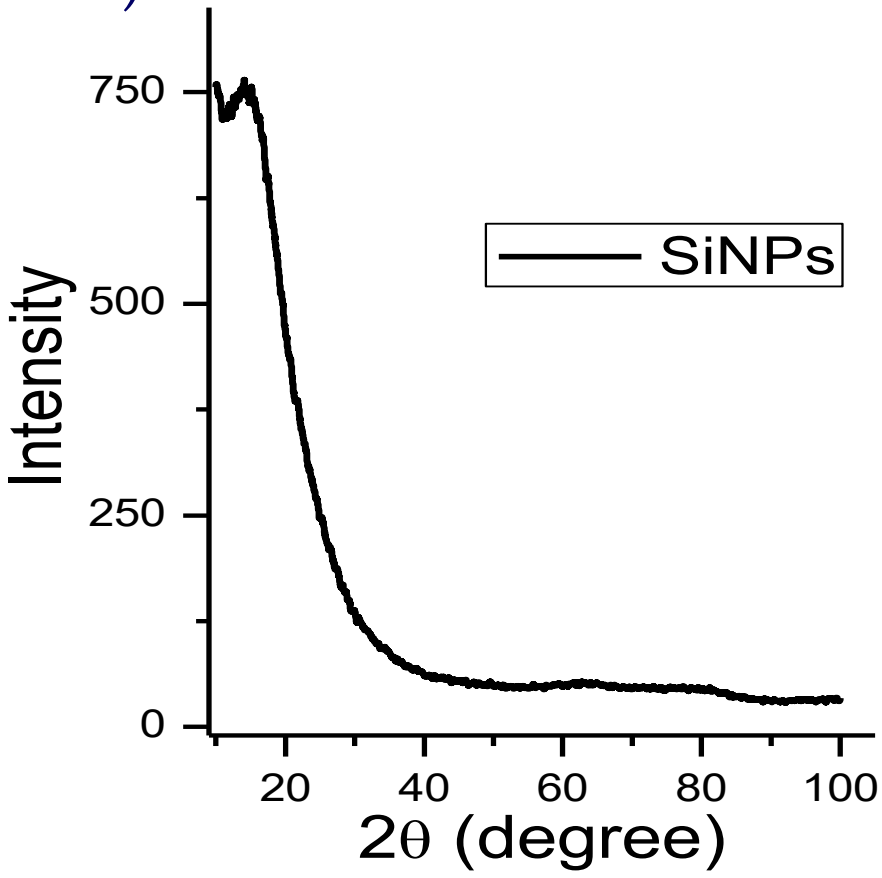
Factors affecting diffraction pattern



Information in a Diffraction Pattern

- Phase Identification (Structure, using Miller indices) & quality ("residual" stress)
- Crystal Size (using Debye-Scherrer equation)
- Texture (crystalline or amorphous)





Strengths

- Powerful for sample identification
- Requires minimal sample preparation
- Relatively straightforward data interpretation

Limitations

- Homogeneous and single-phase material required for sample identification
- Access to a standard reference file of inorganic compounds is required
- For mixed materials, detection limit is » 2% of sample
- For unit cell determinations, indexing of patterns for nonisometric crystal systems is complicated
- Peak overlay may occur and worsens for high angle "reflections"

Acknowledgements

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