X-ray Diffraction (XRD)

How X-rays reveal whether atoms are ordered, and what they are arranged into

- Glass
- Polymers
- Single crystals
- Phosphors
- Ceramics
- Metals
- Nanomaterials
- Glass-ceramics

Varnakavi

What question does XRD answer?

1

Is it ordered?

Crystal (repeating) or glass / amorphous (no repeat)

2

Which material is it?

Each crystal structure has its own fingerprint

3

How big, how strained?

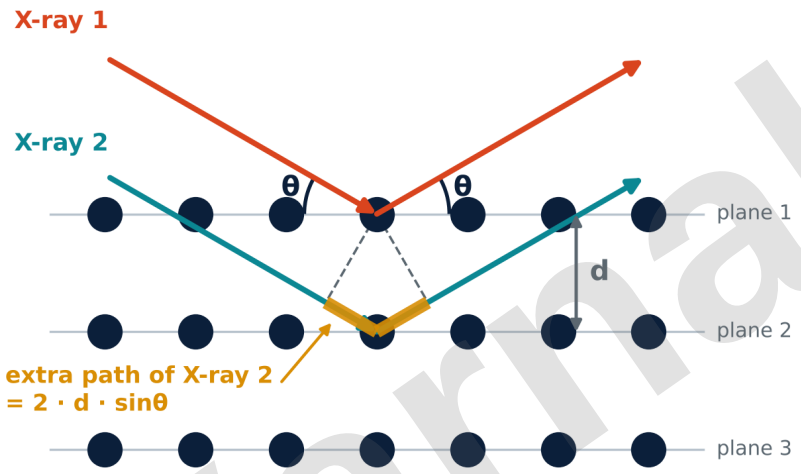
Domain size, strain, texture, crystallinity

1.54 Å

wavelength of the usual Cu X-rays: about the same as the gap between atoms (0.154 nm)

Varnakavi

Bragg's law: when waves line up



Waves reinforce when the extra path is a whole number of wavelengths:

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

Planes: atoms sit in stacked rows, like shelves

Bounce: each row reflects a little X-ray

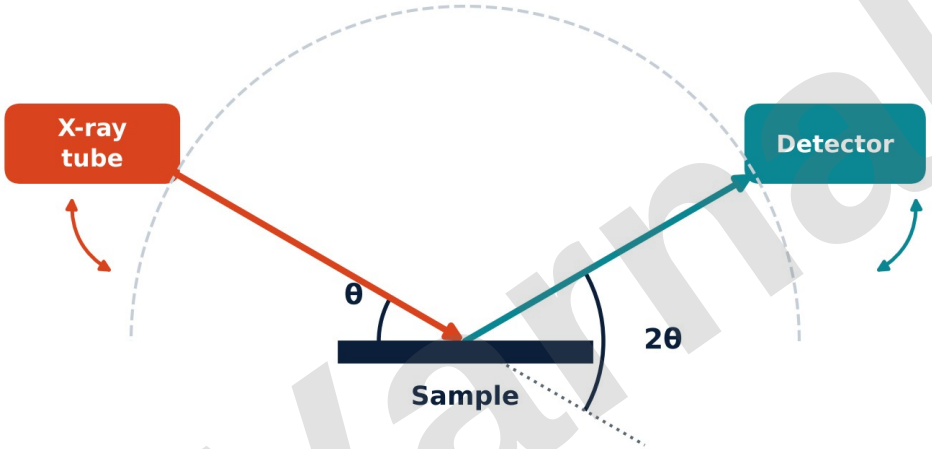
Extra path: the lower ray travels 2 d sinθ further

Match: only at special angles do the waves add up

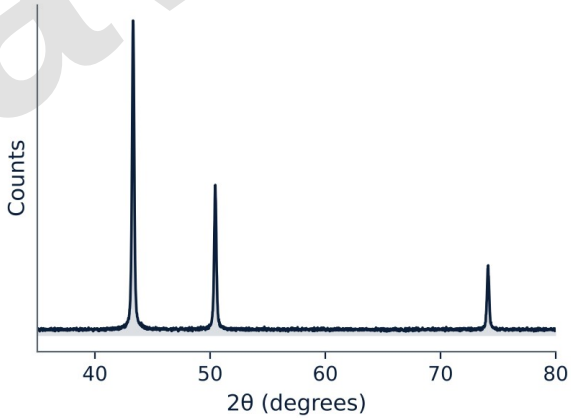
Varnakavi

The instrument: tube, sample, detector

Tube and detector swing
through the angles



The pattern builds up angle by angle



1 Detector counts X-rays at each 2θ

2 Peaks appear where Bragg's law is met

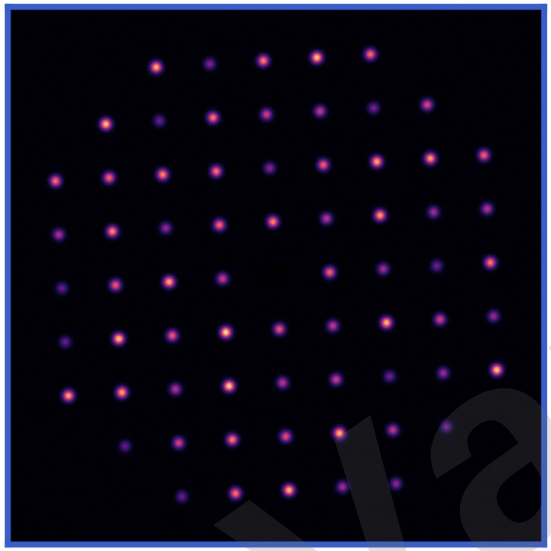
3 Peak positions fingerprint the material

Geometry sets which parts move. Intensity versus 2θ gives the pattern; mounting and optics depend on the sample.

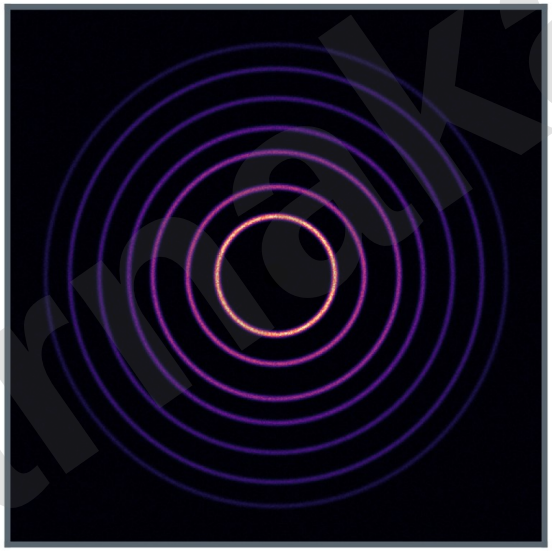
Vanakavi

Three kinds of order, three pictures

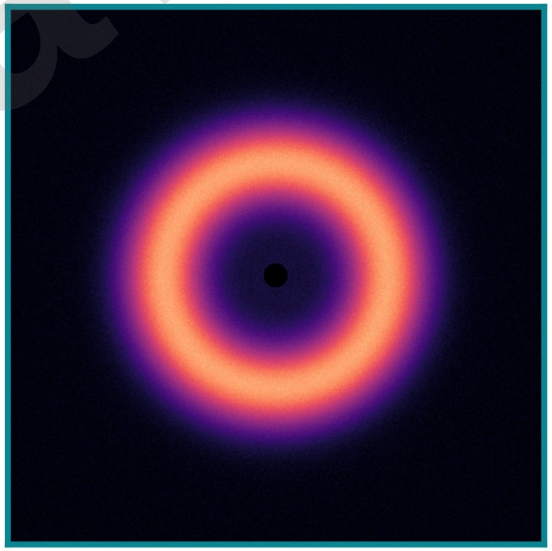
Single crystal
sharp spots: one orientation



Powder / polycrystal
sharp rings: all orientations



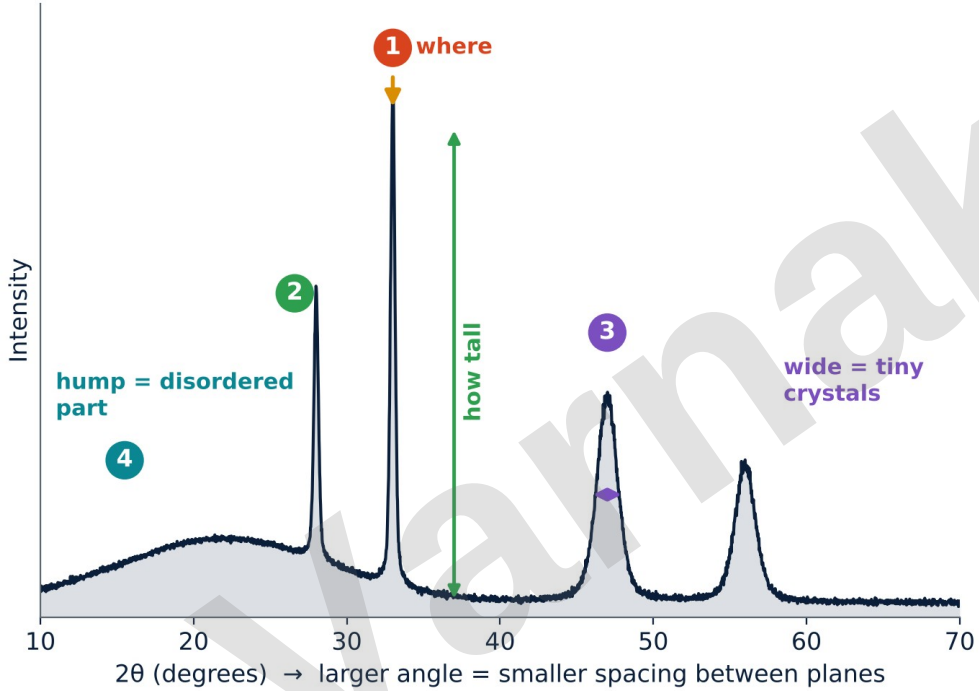
Glass / amorphous
one fuzzy ring: no long-range order



Integrating powder rings gives sharp peaks; glass gives diffuse features. Single-crystal spots depend on orientation.

Vamakavi

Reading a pattern: four clues



1 Peak position

Spacing between atomic planes: unit-cell size, which phase

2 Peak height

Which atoms sit where; also how the grains are oriented

3 Peak width

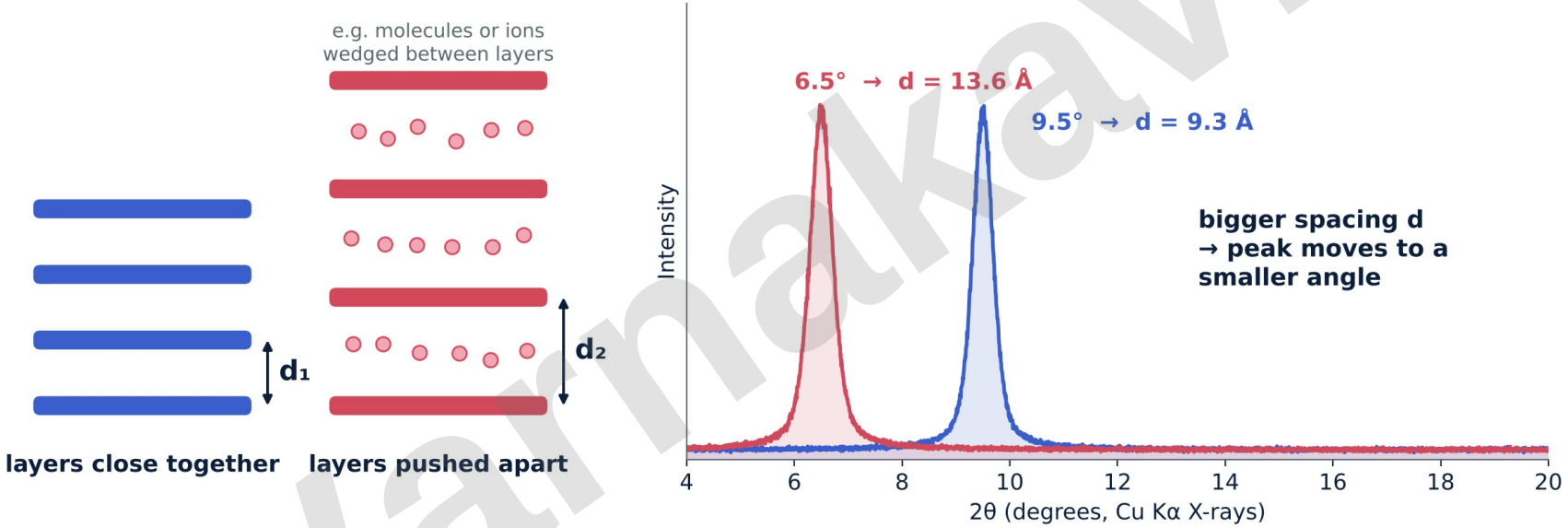
Crystal size (small = broad), plus strain in the lattice

4 Broad hump

Disordered part: glass, polymer chains, amorphous phases

Vanakavi

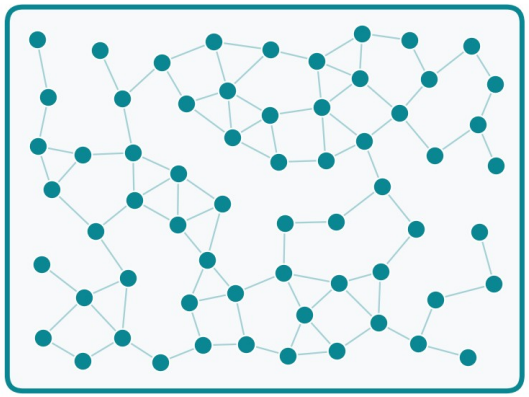
Bragg's law at work: peaks that move

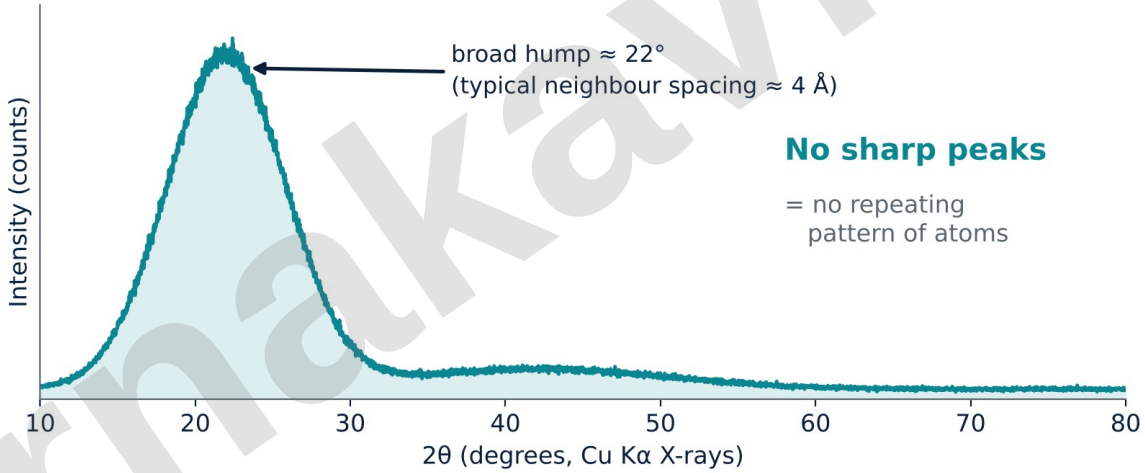


For n = 1 and λ = 1.54 Å: 2θ = 9.5° gives d ≈ 9.3 Å; 2θ = 6.5° gives d ≈ 13.6 Å. Use θ = ½(2θ).

varnakavi

Glass: the missing peaks





Pattern

One broad hump, no sharp peaks

Meaning

Local order remains; long-range periodicity is absent

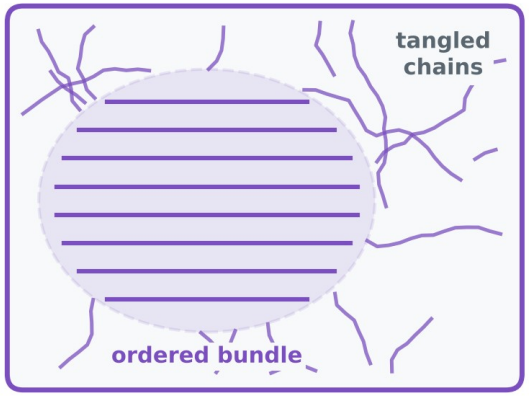
Use it to

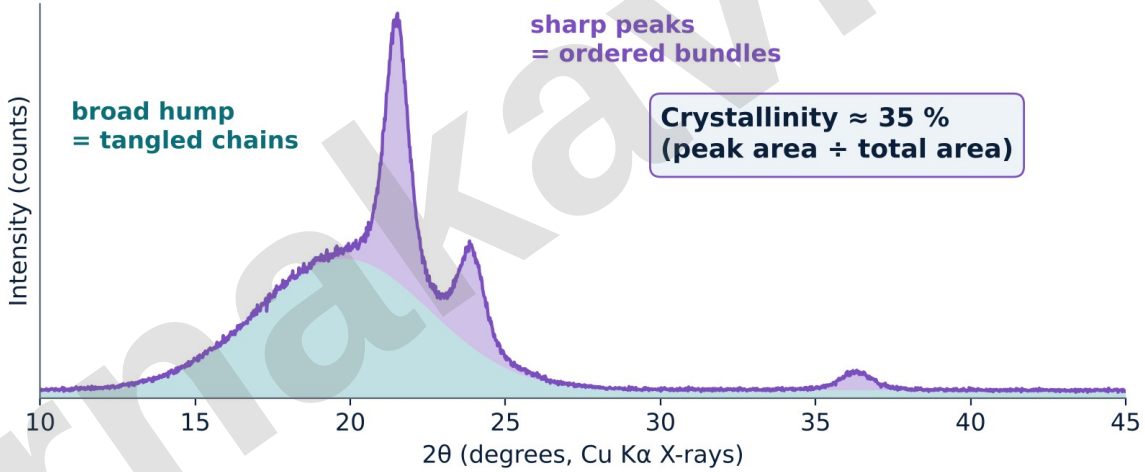
Assess amorphous character within detection limits

Schematic halo; its position is not a nearest-neighbour bond length

Varnakavi

Polymers: ordered and tangled





Pattern
Sharp peaks sitting on a broad hump

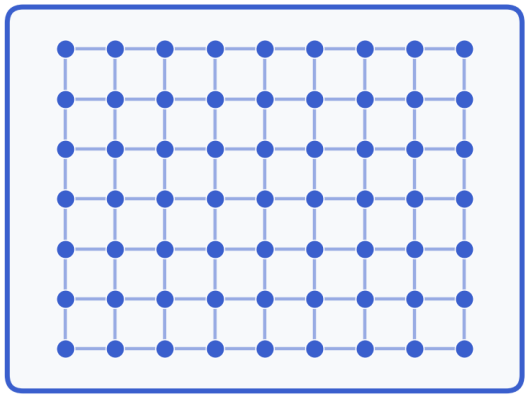
Meaning
Ordered chain bundles inside tangled chains

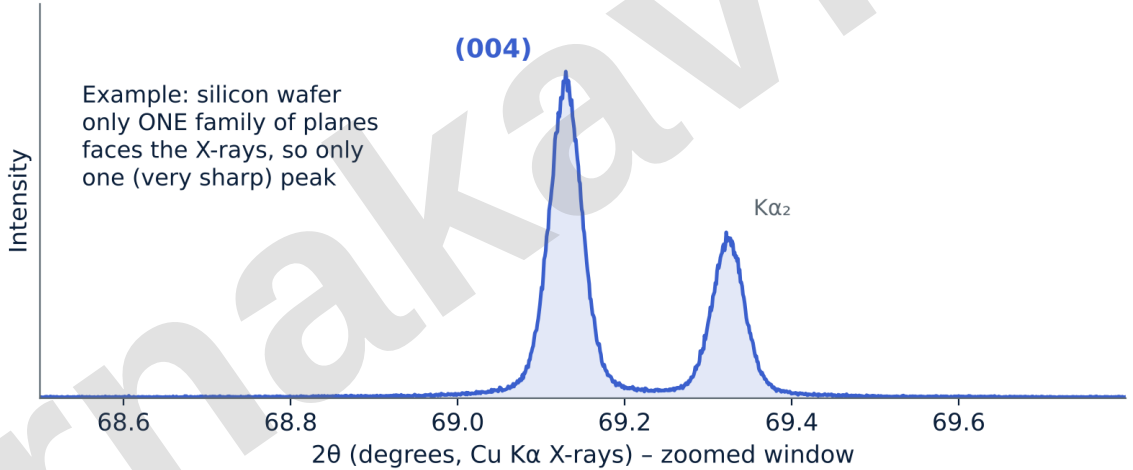
Use it to
Estimate % crystallinity (affects stiffness, clarity)

Simulated pattern for illustration

Varnakavi

Single crystals: very few peaks





Pattern

One or a few extremely sharp peaks (or spots)

Meaning

Symmetric scan selects planes parallel to the surface

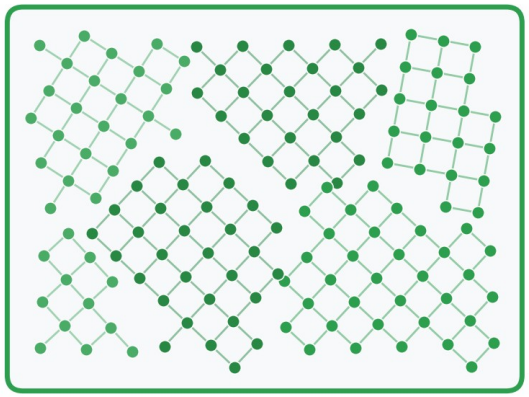
Use it to

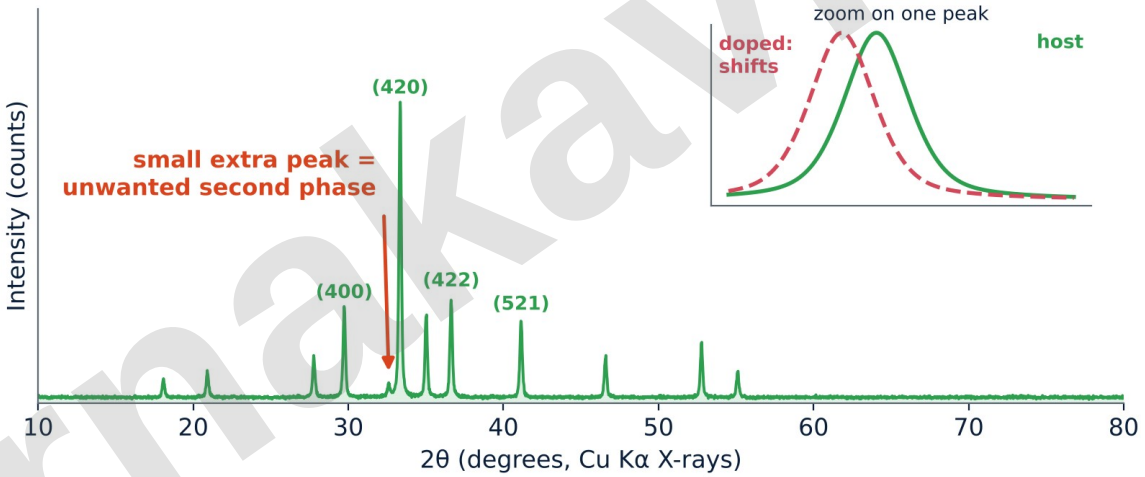
Check orientation, quality and strain of a wafer

Simulated pattern for illustration

Varnakavi

Phosphors: the right crystal





Pattern

Many sharp peaks; compare with a reference card

Meaning

Host structure; shifts alone do not prove doping

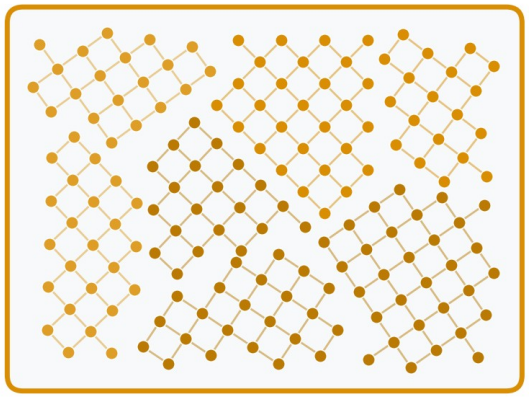
Use it to

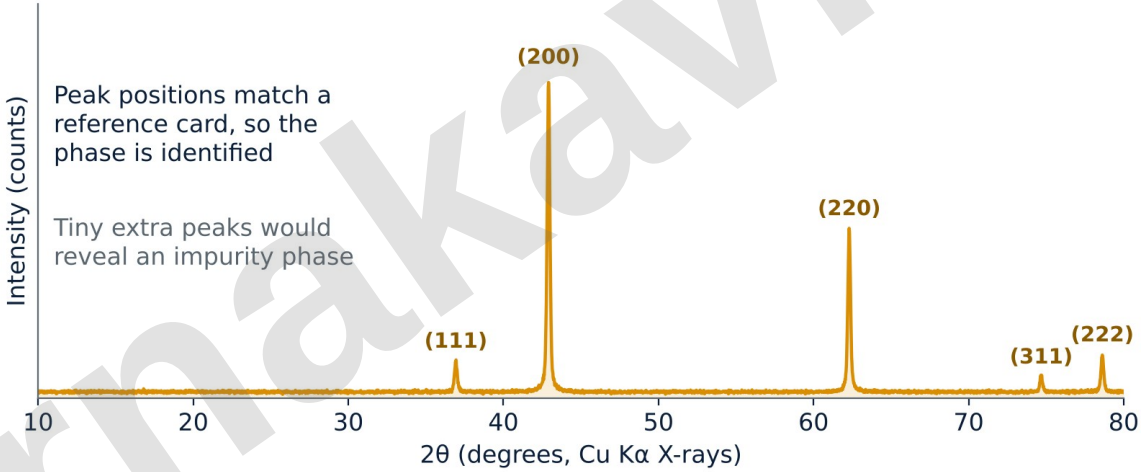
Check phase purity; doping shifts peaks a little

Simulated pattern for illustration

Varnakavi

Ceramics: phase fingerprints





Pattern

Sharp peaks at fixed positions

Meaning

Each crystalline phase has its own fingerprint

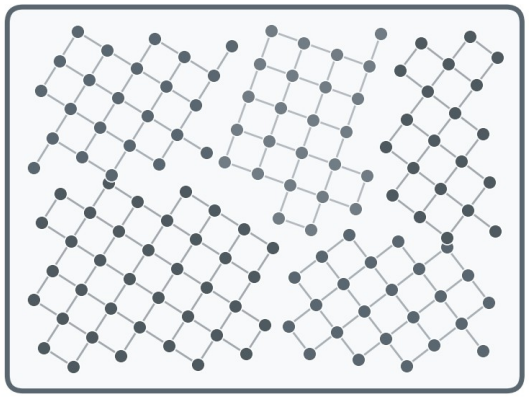
Use it to

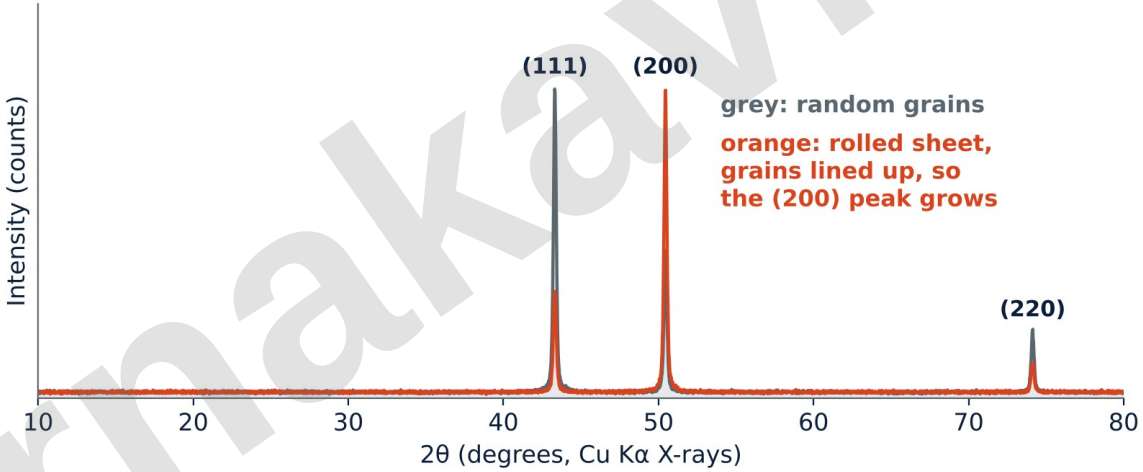
Identify phases and spot impurities

Simulated pattern for illustration

Varnakavi

Metals: lattice and grain direction





Pattern

A few sharp peaks; positions set by the lattice

Meaning

Peak heights depend on how grains are oriented

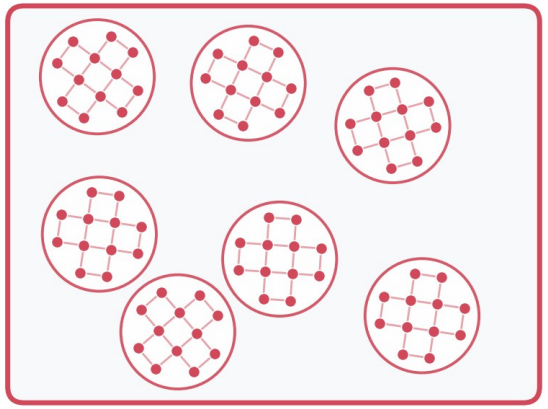
Use it to

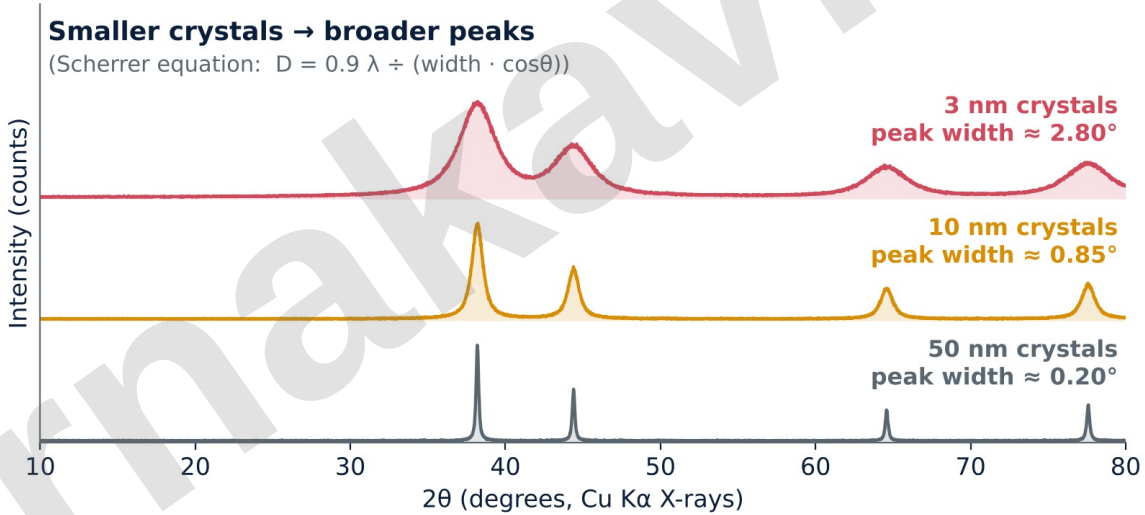
Lattice size, texture and strain from rolling or heat

Simulated pattern for illustration

Varnakavi

Nanomaterials: small means broad





Pattern

Same peak positions, but much broader

Meaning

Small coherent domains can broaden peaks

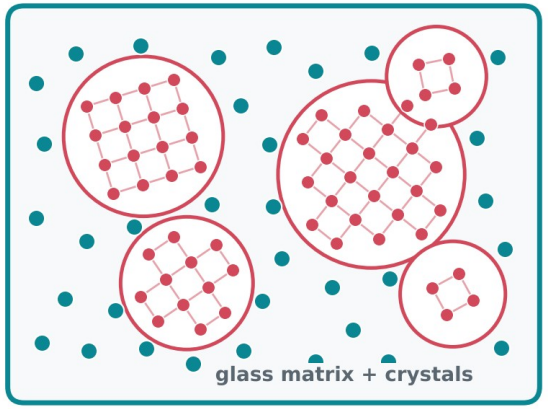
Use it to

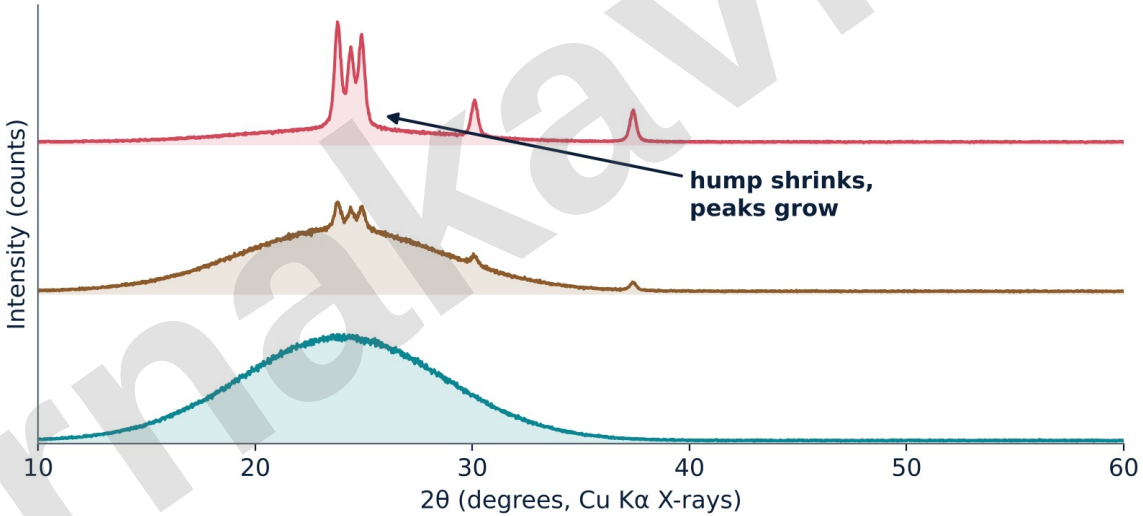
Scherrer: corrected β in radians; domain size

Schematic widths; use corrected β in radians for coherent domain size

Varnakavi

Glass-ceramics: crystals in glass





Pattern
Peaks on top of a leftover hump

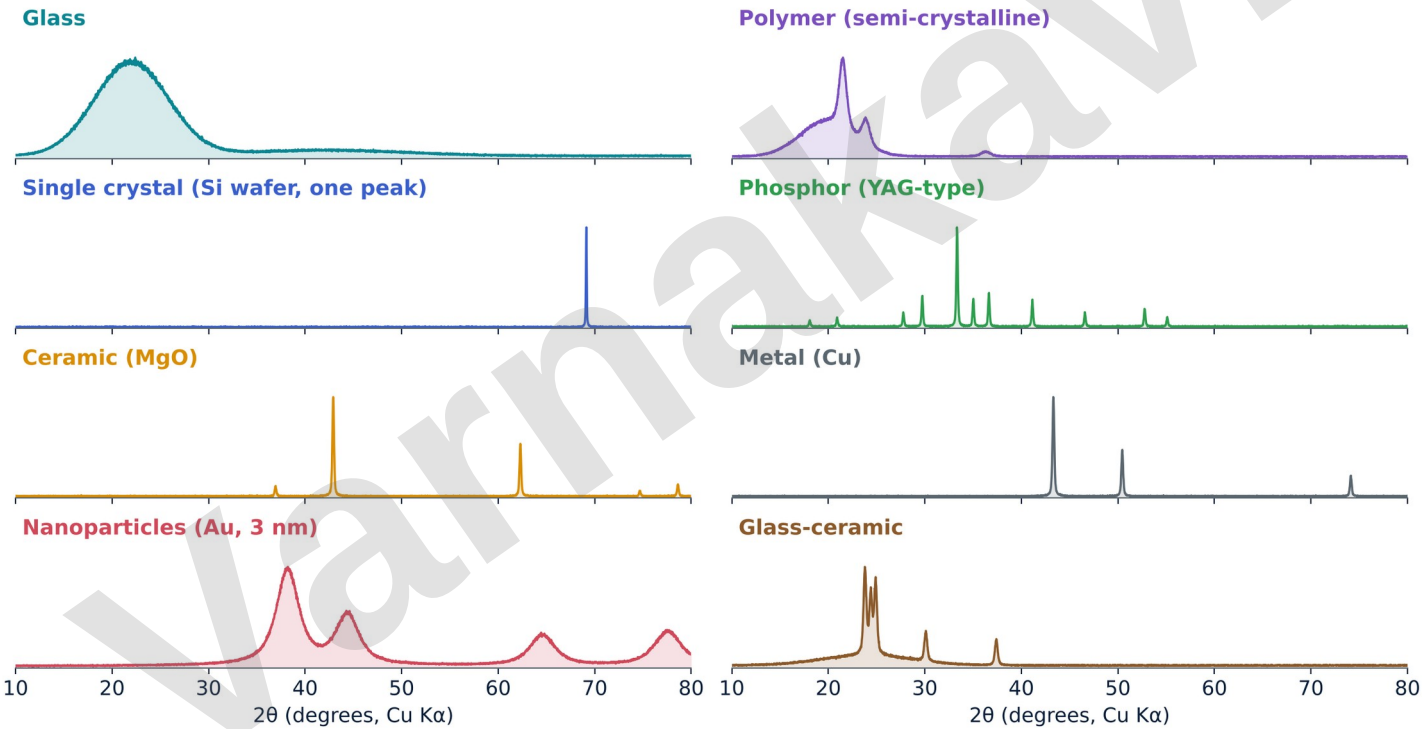
Meaning
Crystals grown inside a glass by heat treatment

Use it to
Track crystallisation and the crystal-to-glass ratio

Simulated pattern for illustration

Varnakavi

All together: one-glance guide



Varnakavi

All patterns simulated for teaching (Cu Kα, 10° to 80° 2θ)

What XRD cannot tell you easily

1 Small amounts hide

Detection depends on sample, optics and counting

2 Light atoms

Hydrogen and lithium scatter very weakly

3 Preferred orientation

Plate-like grains distort peak heights

4 Sample height error

A misplaced surface shifts every peak

5 Overlapping peaks

Phases or broad peaks can mask each other

6 Averages only

It reports the bulk average, not one particle

Varnakavi

Takeaways

1. Bragg's law links peak angle to atomic spacing
2. Sharp peaks mean order; a broad hump means disorder
3. Size, strain and instrument effects broaden peaks
4. Peak positions fingerprint the phase

Varnakavi