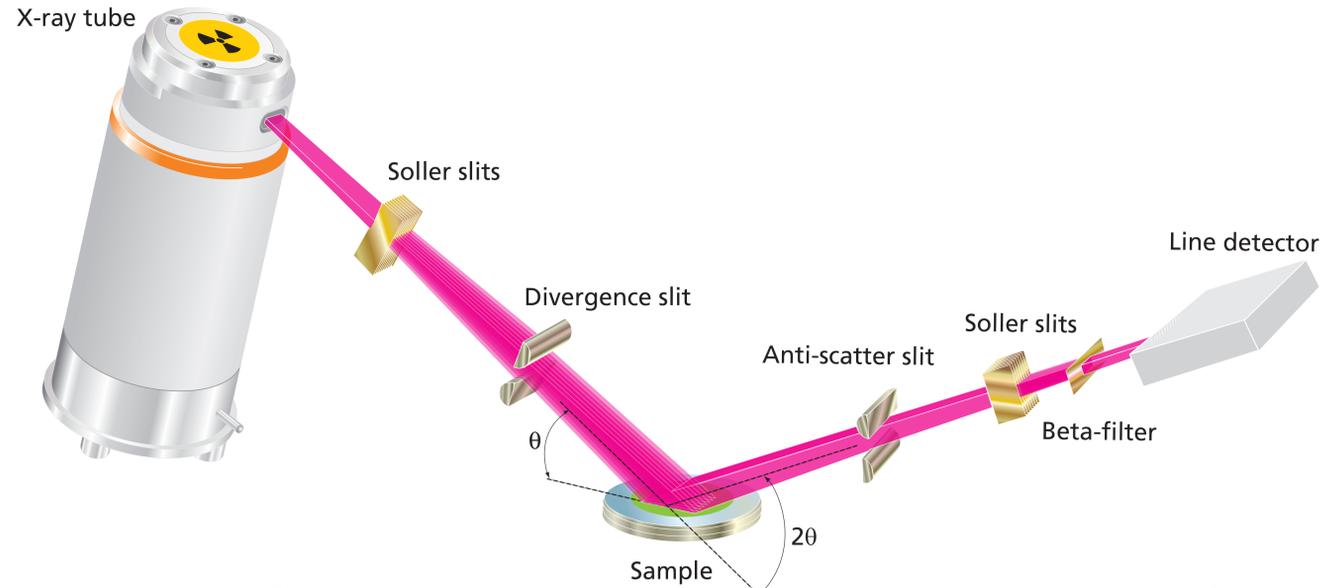


X-RAY POWDER DIFFRACTION

What is X-ray diffraction?

X-ray diffraction is a versatile, non-destructive analytical technique for identification and quantitative determination of the various crystalline forms, known as 'phases', of compounds present in powdered and solid samples. Identification is achieved by comparing the X-ray diffraction pattern - or 'diffractogram' - obtained from an unknown sample with an internationally recognized database containing reference patterns for more than 500,000 data sets.

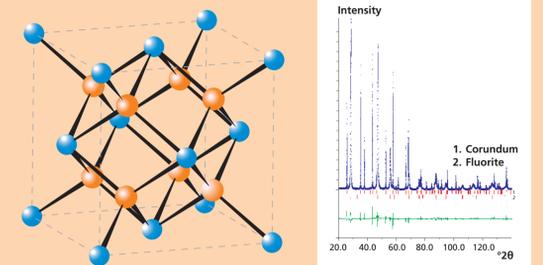
Modern computer-controlled diffractometer systems use automatic routines to measure, record and interpret the unique diffractograms produced by individual constituents in even highly complex mixtures.



What is the structure of the crystals?

A crystal structure is a periodic arrangement of atoms. It is composed of a three-dimensional crystal lattice and a motif, a spatial arrangement of a set of atoms. The crystal structure can be described in terms of unit cells, stacked according to the points of the crystal lattice.

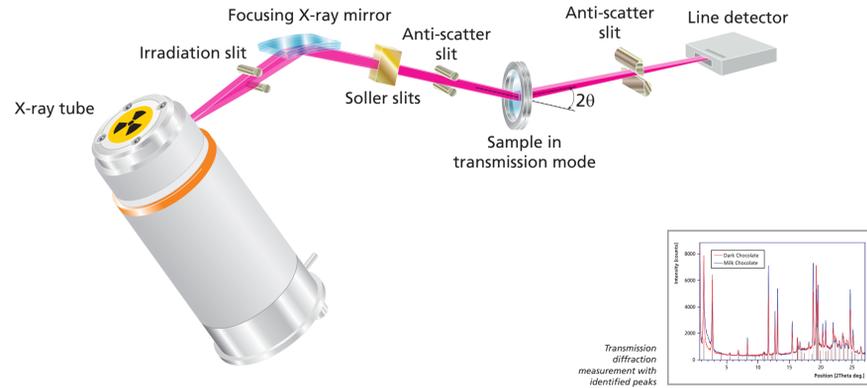
The Rietveld method refines a crystal structure by comparing the measured diffraction pattern with that calculated from a known crystal structure. A least-squares refinement is used to optimize the structure parameters.



Which phases are present?
At what concentration levels?
What is the amorphous content of the sample?

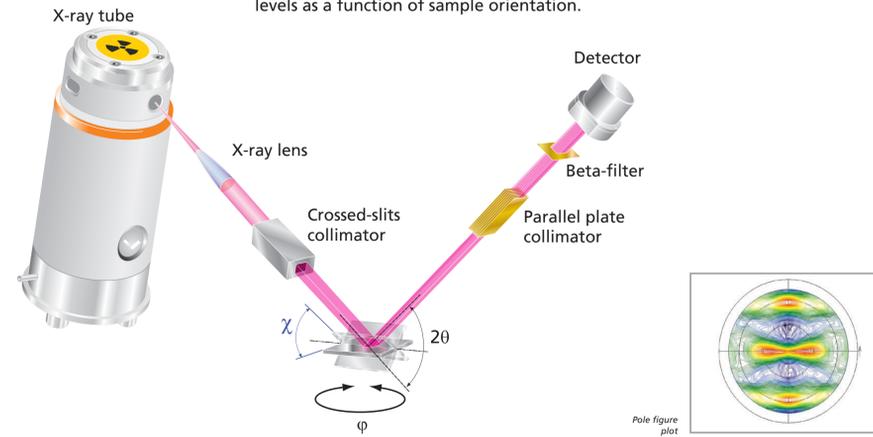
Transmission

Transmission diffraction is used to analyze X-ray transparent materials, such as pharmaceuticals, for low-angle powder diffraction or for investigating preferred orientation issues. Typically the sample is prepared between transparent foils. The resulting scans can be used for phase identification and crystallography analysis.



Texture

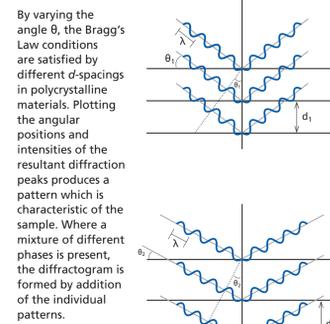
Measurement of texture – the non-random, or preferred, orientation of crystallites – involves measurement of the variations in intensity of a single Bragg reflection as the sample is both tilted (χ) and rotated (ϕ). The result is plotted as a 'pole figure', in which the contours indicate intensity levels as a function of sample orientation.



How does it work?

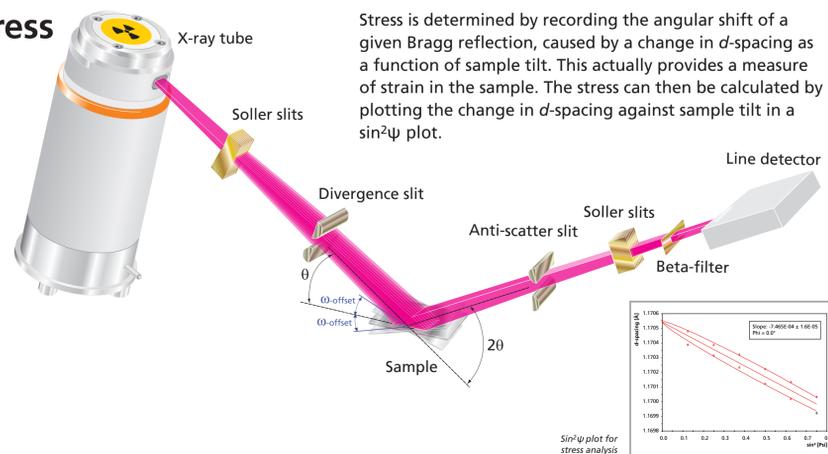
Crystal lattice
A crystal lattice is a regular three-dimensional distribution (cubic, rhombic, etc.) of atoms in space. These are arranged so that they form a series of parallel planes separated from one another by a distance d , which varies according to the nature of the material. For any crystal, planes exist in a number of different orientations – each with its own specific d -spacing.

Diffraction
Bragg's Law: $n\lambda = 2d \sin\theta$
When a monochromatic X-ray beam with wavelength λ is incident on lattice planes in a crystal at an angle θ , diffraction occurs only when the distance travelled by the rays reflected from successive planes differs by a complete number n of wavelengths.



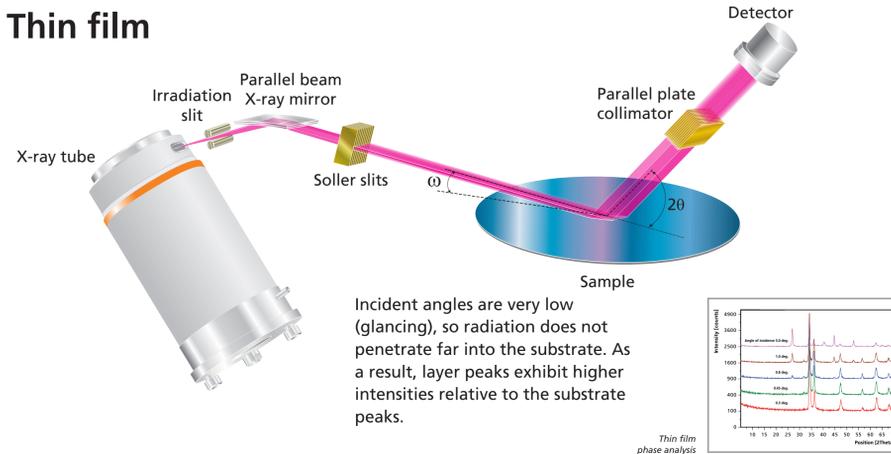
Stress

Stress is determined by recording the angular shift of a given Bragg reflection, caused by a change in d -spacing as a function of sample tilt. This actually provides a measure of strain in the sample. The stress can then be calculated by plotting the change in d -spacing against sample tilt in a $\sin^2\psi$ plot.



Thin film

Incident angles are very low (glancing), so radiation does not penetrate far into the substrate. As a result, layer peaks exhibit higher intensities relative to the substrate peaks.



The sample
The diagram depicts a typical sample, comprising two crystalline phases (violet, blue), each with different average crystallite sizes, plus a proportion of amorphous material (beige). The features of the diffractogram shown below are colour coded to indicate the relevant components.

Crystallite size
The widths of the peaks in a particular phase pattern provide an indication of the average crystallite size. Large crystallites give rise to sharp peaks, while the peak width increases as crystallite size reduces.

Peak broadening also occurs as a result of variations in d -spacing caused by micro strain. However, the relationship between broadening and diffraction angle 2θ is different from that of crystallite size effects, making it possible to differentiate between the two phenomena.

The result
The result of an XRD measurement is a diffractogram, showing phases present (peak positions), phase concentrations (peak areas), amorphous content (background hump) and crystallite size/strain (peak widths).

