UNIVERSITY OF LIMERICK
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Bernal Institute

X-ray Diffraction (XRD)
PANalytical

Overview

X-ray Diffraction is an analytical technique that uses an inherent property of the x-ray beam i.e. the wavelength and the laws of physics, that determine how that beam interacts with matter, to characterize materials. Crystalline materials are characterized by the long- range orderly periodic arrangements of atoms. The technique is applied primarily to determine crystal structures, identify phase composition, measure stress, preferred orientation and crystallinity. Bragg's law calculates the angle where constructive interference from X-rays scattered by parallel planes of atoms will produce a diffraction peak. Only a small fraction of the crystallites in the specimen actually contribute to the measured diffraction pattern.

XRD Capabilities

- Inorganic phase analysis on powder even on very small sample quantities. Analysis of polymorphs.
- In situ phase transformations. Crystallisation phenomena as a function of temperature can be studied at temperatures up to 1200^{o}C
- Measuring of the thickness of poly-crystalline thin layers and interpretation of the roughness at the layer interface
- Crystallographic measurements, thin-film thickness measurement
- Determination of crystal parameters by diffraction of X-rays
- Texture Analysis – to determine the preferred orientation of the crystallites in polycrystalline aggregates
- Residual stress – based on the measurement of lattice strain distributions. Stress is calculated from strain distribution.

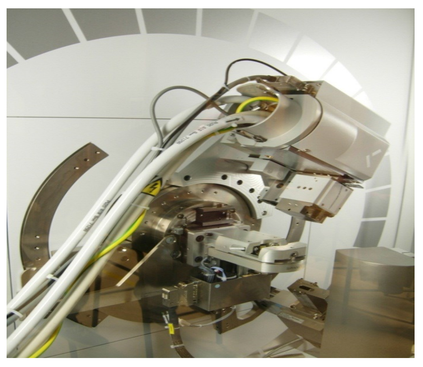
Technical Details

The Bernal Institute has two PANalytical Diffractometers, (i) X'Pert and (ii) Empyrean for the analysis of powders, thin films, nano-materials and solid objects using a Copper anode. Powder samples can be run in reflection or transmission mode. Different applications are available on each diffractometer. Non ambient temperature stages allow the investigation of phase change with temperature.

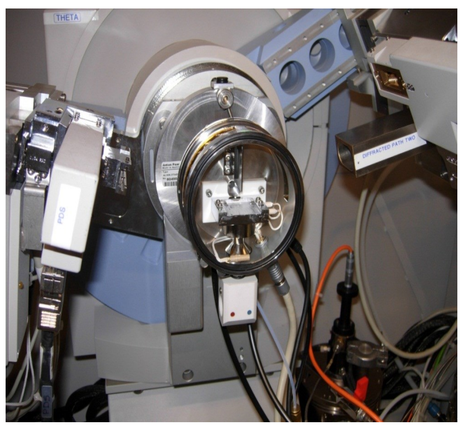
PANalytical (i) X'Pert and (ii) Empyrean Diffractometers.

Transmission X Ray Diffraction for Pharmaceutical Powders
Rather than reflecting off the upper surface of the sample, the X ray passes through it. The recognised method which normally gives the best results for investigating organics like pharmaceuticals is the transmission geometry with parallel X-ray beam.

Measuring film thickness on chi-phi platform on Empyrean

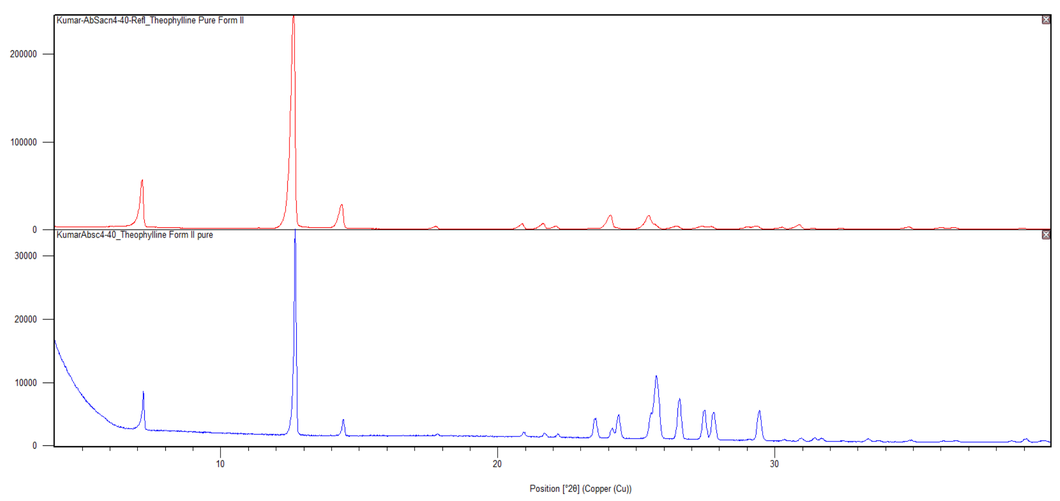


Empyrean set in transmission mode for PXRD of very small samples



TTK 450 Low temperature stage (-180°C to 450°C) with cryo unit for phase change in metals, pharmaceuticals etc.

A wide range of sample sizes and shapes and applications e.g. stress, texture



Theophylline Form II sample show in reflection (top, red) and transmission (bottom, blue)

Analysis

High Score software
•Qualitative phase analysis.
•Crystallinity determination.
•Crystallite size or micro strain calculations by the Scherrer method.
•Quantitative phase analysis using Rietveld

Contact

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