

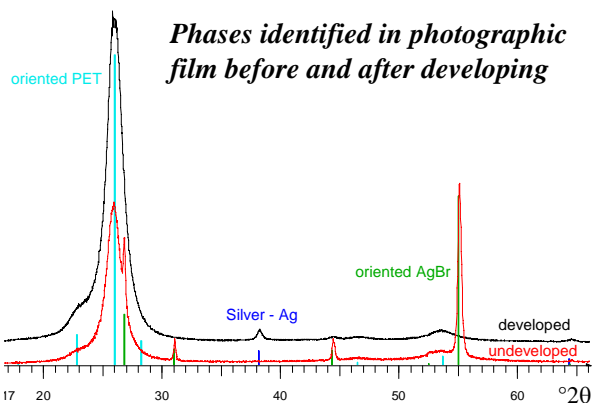
X-Ray Diffraction

Most of the materials we use are partly or completely crystalline and the nature of that crystallinity has a major effect on their properties. X-ray Diffraction (XRD) is the primary tool for characterising crystallinity and also has applications for amorphous materials. It can be applied to solids, powders, colloids and liquids. We use XRD to obtain a range of information:

- Identification of crystalline phases
- High temperature and reactive atmosphere studies
- Microstructure: crystallite size and shape, lattice distortion, faulting
- Crystallinity
- Crystal structure variations, e.g. solid solutions and polymorphs
- Orientation: crystalline and amorphous
- Non-crystalline periodicity and size

Phase Identification

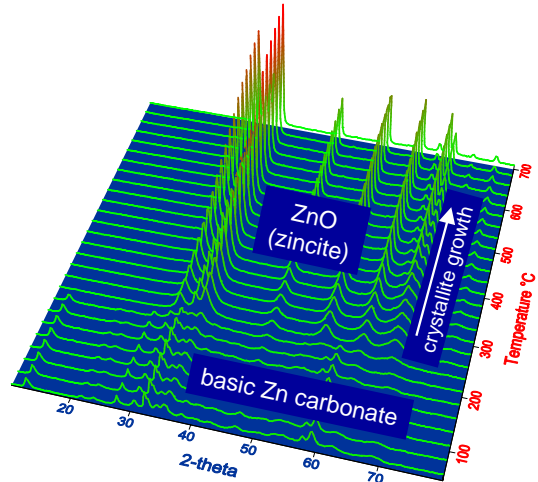
Identifying the crystalline phases in a material is a very traditional use of XRD and fundamental to understanding properties. It goes beyond elemental analysis to differentiate on the basis of geometric structures. The diffraction patterns of photographic emulsions below show the transformation of AgBr to Ag metal and that the base film is biaxially oriented PET (polyethylene terephthalate). It can also be deduced that the AgBr crystallites are oriented and platey in shape. The Ag metal crystals are smaller and both phases contain solid solution impurities.



Depth profiling by grazing incidence XRD can also be used to study the emulsion structure.

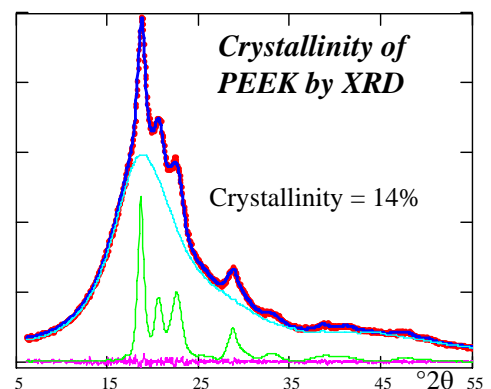
Non-Ambient XRD

In situ measurements are important to understand what really happens as materials are processed and can reduce the total cost of exploring different conditions. We have a range of equipment for these studies that makes it possible to obtain most types of XRD information under variable temperature and reactive environment conditions. The example below shows the thermal transformation of a basic zinc carbonate to zinc oxide. Detailed analysis reveals the development of crystallite size (including size distribution) and shape in addition to reaction kinetics.



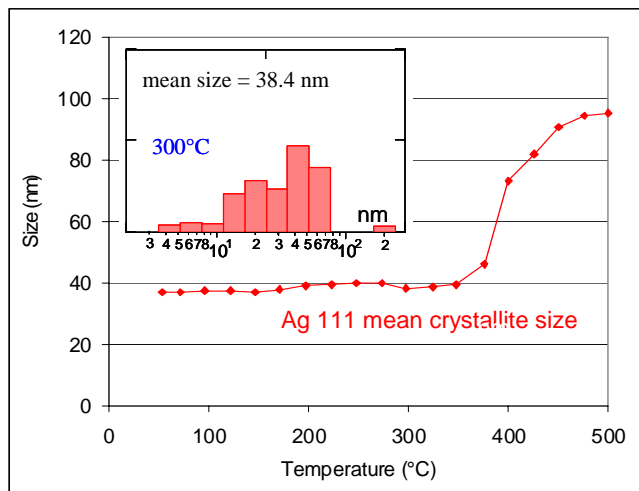
Crystallinity

The degree of crystallinity affects many material properties and XRD is the most adaptable means of measuring it. It can be applied to materials from polymers to glass ceramics.



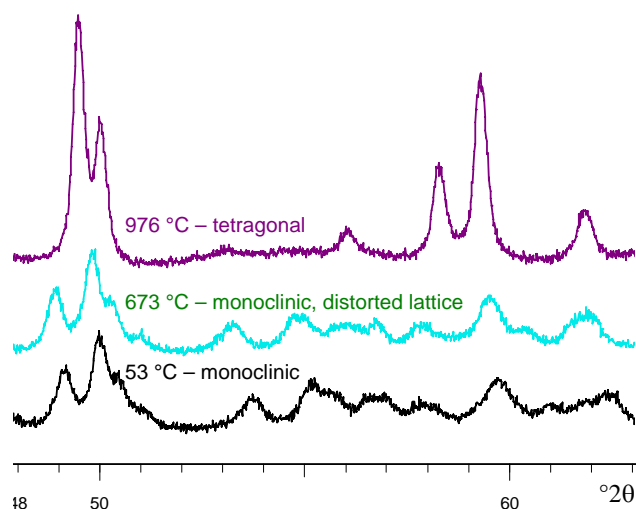
Microstructure

Microstructural information from XRD peak shapes includes crystallite size, size distribution, shape, lattice distortion and faulting. These characteristics are relevant to properties such as chemical reactivity (as in the data for the Ag catalyst below), mechanical strength and toughness, processing characteristics, etc.



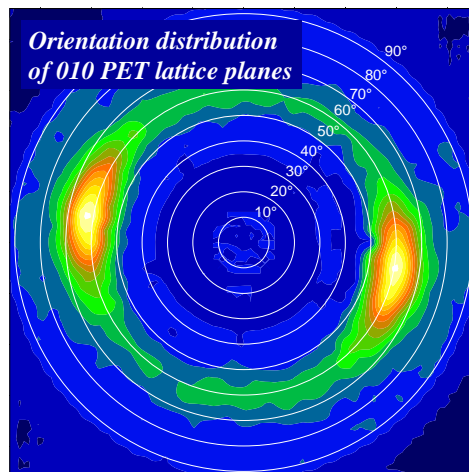
Crystal Structure Variations

Diffraction patterns are sensitive to modifications of crystal structure, e.g. introduced by impurities in solid solution. The illustration below shows the effect of temperature on the lattice parameters of monoclinic zirconia and the higher temperature transformation to the tetragonal form. These variations can be followed in terms of lattice parameters and atomic positions.



Orientation

The orientation of crystallites within materials affects the mechanical, optical and conductive properties. The 'pole figure' below shows the angular distribution of PET crystallites in a film. This orientation was produced during the film drawing process. It is also possible to measure amorphous orientation.



Small Angle X-Ray Scattering (SAXS)

To measure larger spacings, such as polymer long periods or from heavily intercalated clays, special SAXS instrumentation is required. The technique is also useful for measuring sizes and shapes (can be non-crystalline) up to about 200 nm. This is often the method of choice for comparing colloidal structures.

Capability

- We have three Bruker Diffractometers including:
- High temperature / environmental (to 1600°C) stages for working with solids and liquids.
 - Eulerian cradle for orientation measurements.
 - Reflection, transmission and capillary geometry
 - Grazing incidence
 - Parallel beam geometry for irregular samples
 - Reflectometry
 - Kratky SAXS with temperature control

To learn more about XRD in MSG and how it can be applied to your interests, please contact Dr Steve Norval:

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