

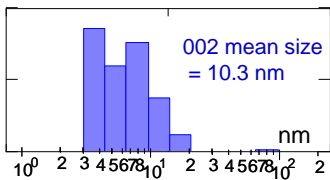
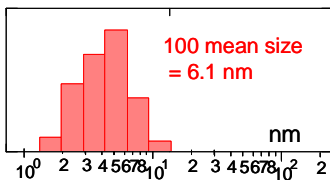
## X-Ray Diffraction – a tool for nano-technology

### Introduction

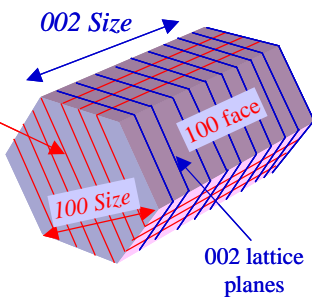
The wavelength of X-rays is on the atomic scale, so X-ray diffraction (XRD) is a primary tool for probing structure of nano-materials. Crystalline materials give rise to the most obvious applications, but there is also important information to be obtained from semi-crystalline and even amorphous materials.

### Crystallite Dimensions

Crystallite size is a fundamental characteristic of nano-materials and microstructure (call it “nanostructure” if you like) is often a key to understanding and controlling bulk properties. Small crystallite size results in broadened diffraction patterns. Analysis of peak shapes can give information about crystallite size and other aspects of microstructure, particularly lattice distortions (due to variations in composition or micro-strain) and faulting.



### Size and Shape of ZnO crystallites



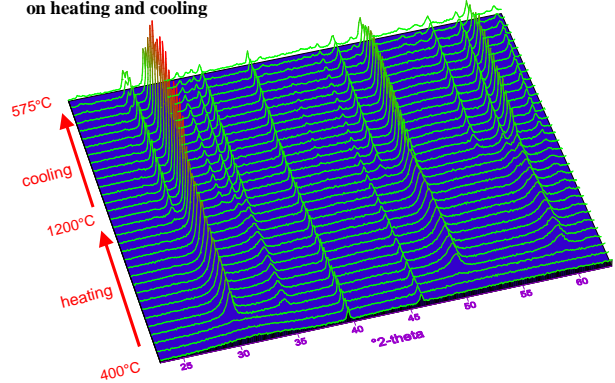
We analyse line broadening using a simulation and refinement method that gives crystallite size distributions independent of any mathematical function to fit peak shapes. This means that multimodal distributions can readily be characterised. Since this broadening is related to the geometry of lattice planes within the crystallites, it is often possible to determine shape parameters by analysing multiple lines, as illustrated above.

### Crystalline Phases & Structures

The most traditional use of XRD is still one of the most important – to identify the crystalline forms, or the phases that are present. In materials ranging from polymers to glass ceramics, the degree of crystallinity is a fundamental characteristic and XRD is the most adaptable means of measuring it. More detailed information about nature of the crystallinity can often be obtained by refining crystal structure parameters

as a basis for structure/property relationships, e.g. in relation to the formation of solid solutions. The diagram below follows phase changes in a finely crystalline zirconia from an amorphous starting material through crystal form changes on heating and cooling.

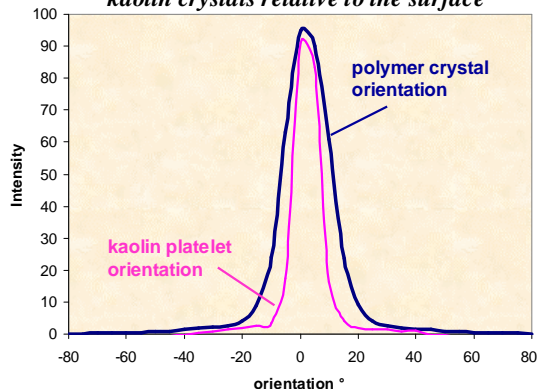
*In situ* XRD of zirconia, showing phase changes on heating and cooling



### Orientation

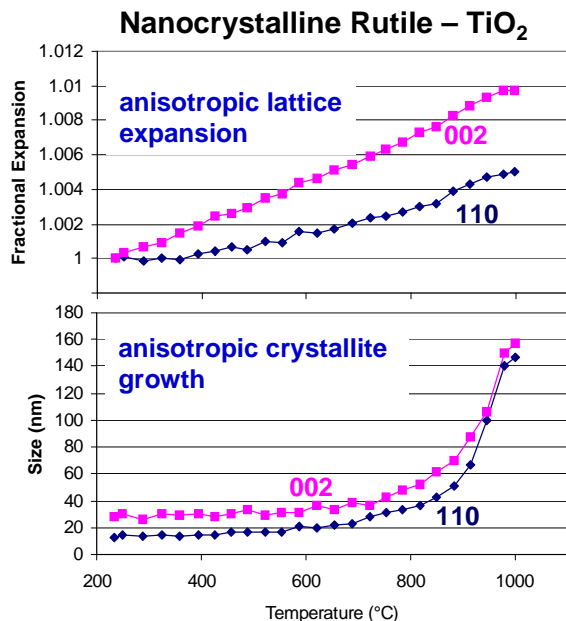
The crystallites in a fabricated structure are often oriented as a design feature or as a characteristic of the processing technology. Either way they can have a big effect on the product performance. The diagram below shows the orientation distribution of tiny platelets of kaolin on the surface of a polymer film. The platelets, only a few nanometres thick, are so well aligned with the surface that they have a major impact on the surface properties of the polymer.

*Orientation of polymer crystals and kaolin crystals relative to the surface*



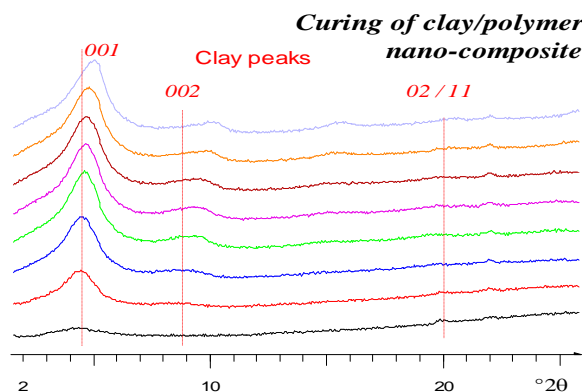
### In Situ Measurement

The ability to make *in situ* measurements is important when trying to understand what really happens during processing and to reduce the total cost of exploring different process conditions. In addition to following the kinetics of the phase changes, a more complete model of the system can be based on changes in the lattice parameters and crystallite sizes of the components.



**Dynamic Phenomena**

Studies can be made under reactive atmospheres and in liquids with rapid measurement of diffraction patterns for real time studies. The diagram below shows the development of orientation and intercalation spacing in a clay / polymer nano-composite during curing of the polymer.



This brochure gives a few examples of the information that we can obtain by XRD about structures on the nano-scale. We are always pleased to bring our wide experience of this work to bear on different materials, either using a tried and tested solution, or by inventing something novel.

**Information from XRD**

- Phase identification and quantification
- Crystal structure variations (e.g. by lattice parameters)
- Crystallite size and shape, and lattice distortion
- Crystallinity
- Non-crystalline periodicity and size
- Orientation (crystalline and amorphous)
- Dynamic studies
- *In situ* studies at process temperatures and in reactive atmospheres

**Examples of materials studied**

Catalysts, ceramics, clays, coatings, colloids, composites, foods, liquid crystals, liquid suspensions, metals, paints, pigments, pharmaceuticals, polymers, nano-composites, nano-inorganics and -organics, semiconductors, surfactants.

To learn more about XRD in MSG and how it can be applied to your interests, please contact Dr Jeff Franks or Barbara Middlemiss:

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If you would like to learn more about MSG's other capabilities and how they might help your business, please address your enquiries to Dr Allan Stewart at the numbers below.

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