Intertek **MSG**

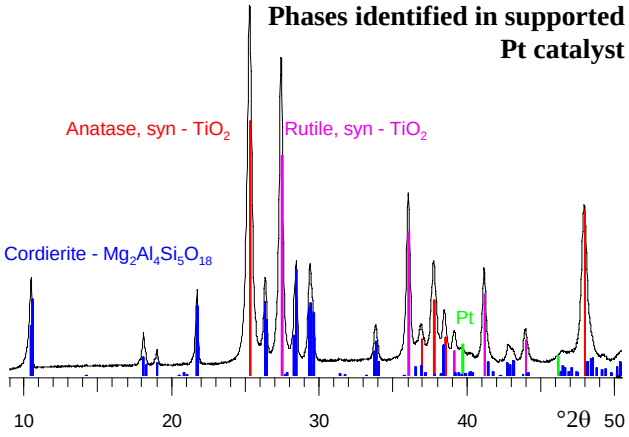
Characterising Catalysts by X-Ray Diffraction

Introduction

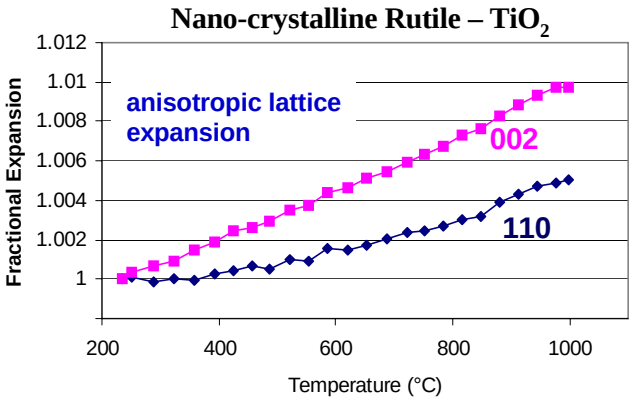
Heterogeneous catalysts typically comprise multiple nano-crystalline components where the crystallite chemistry and physical arrangement provide the basis for the catalytic function. X-ray diffraction (XRD) is well-suited to probing structure on this scale, giving insights into phase composition and microstructure.

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. The diffraction pattern below was used to identify the component phases in a monolithic platinum oxidation catalyst.

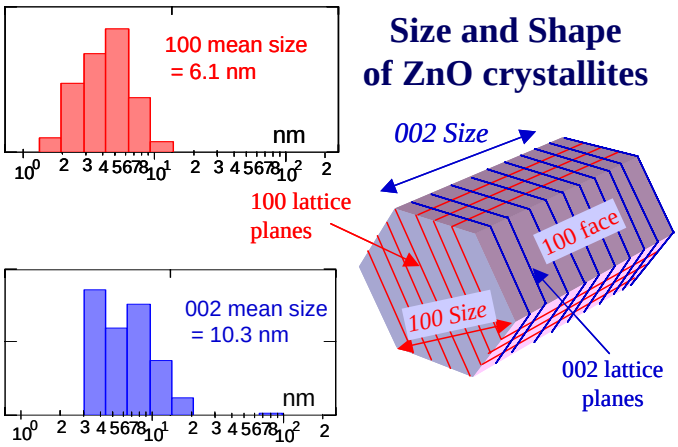


More detailed information about nature of the crystallinity can often be obtained by refining crystal structure parameters as a basis for structure/property relationships, e.g. in relation to the formation of solid solutions. The diagram below follows lattice parameter changes in a finely crystalline rutile as it is calcined.



Crystallite Dimensions

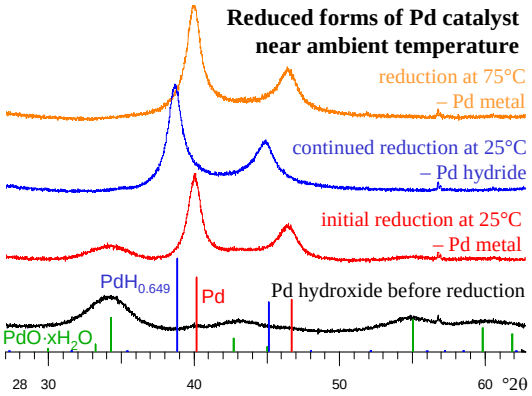
Crystallite size is directly related to surface area and can often be measured for each active or passive component. Size information is derived from the shape of diffraction peaks. Individual peaks relate to different crystal directions and give crystallite shape, as shown below for a zincite support. Other aspects of microstructure, e.g. due to variations in composition, micro-strain or faulting, can also be derived.



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 multi-modal distributions can readily be characterised.

In Situ Measurement

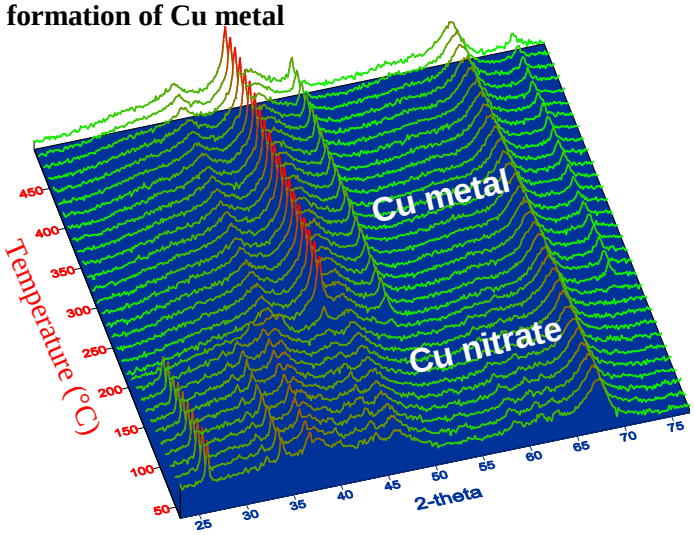
In situ measurements are particularly important when trying to understand what really happens during catalyst processing, conditioning and deactivation, e.g. the active form of a Pd catalyst is highly sensitive to reaction conditions around room temperature:



Characterised by Expertise

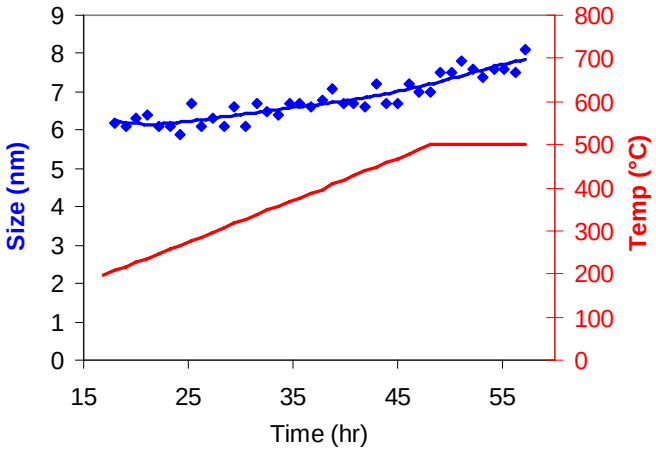
www.measurementscience.co.uk
www.intertek-cb.com

***In situ* XRD reduction of Cu nitrate / alumina catalyst showing the phase change on formation of Cu metal**



Phase changes can readily be followed under reaction conditions. Typical catalyst reduction data is shown above for a Cu nitrate precursor supported on γ-alumina, reducing to Cu metal. There was brief evidence of an intermediate phase. Catalyst sintering studies are illustrated by the subsequent growth of Cu crystallites following the phase transformation (below).

Growth of Cu crystallites in Cu / alumina catalyst following reduction



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 Intertek MSG and how it can be applied to your interests, please contact:

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