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CHEMICAL INDUSTRY FOCUS ISSUE 3

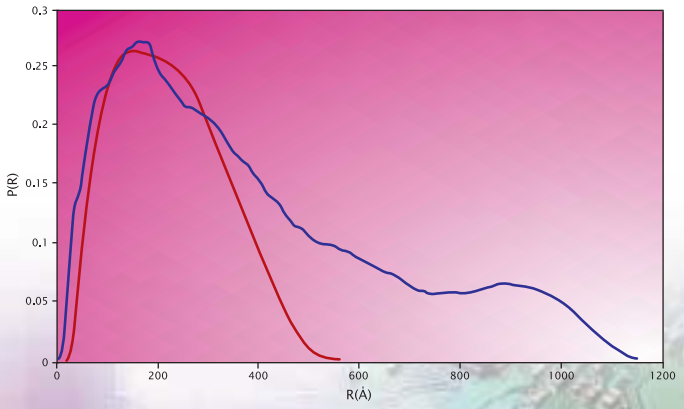
DARTS 'Braves' ChemSpec Europe 2003 and Launches its Nanoparticle Size Distribution Measurement Technology

Welcome to the third edition of Chemical Industry Focus - the *DARTS* newsletter designed specifically for the chemical, speciality and related industries. Thanks to all of you who visited the *DARTS* stand at ChemSpec Europe 2003 and braved the fury of the protesters. If you missed us at G-Mex this newsletter should keep you up to date with what's going on at *DARTS* currently of interest to the chemical and related industries. In this issue we look at how *DARTS* has solved structures in catalytic systems and the ground-breaking work on determining nanoparticle size distributions in the range 1 - 100 nanometres.

Chris Pickles (right) ***DARTS*** Marketing Manager, meets potential customers on the ***DARTS*** stand at ChemSpec Europe 2003, held at G-Mex.

DARTS Cracks the 'Holy Grail' of Nanoparticle Size Measurement

The market for nanopowders is small but growing rapidly. Already there are over 70 producers in the world but they are small scale. Application areas extend from drug delivery to electronics to printing to cosmetics.



One major unsolved problem is particle size determination – particularly particle size distribution measurement in the range 0–100 nm.

DARTS has solved this problem by applying the technique of small angle X-ray scattering under specific experimental conditions and by an advanced data treatment developed at Daresbury.

The figure shows the quality of particle size distribution plots that can be obtained by the method in the size range 0-120 nm (0-1200 Å) for two quite different samples of nanopowder materials.

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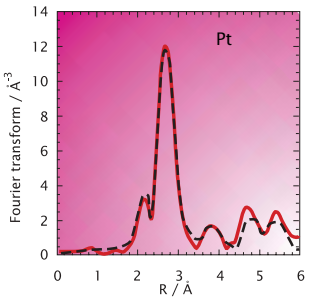
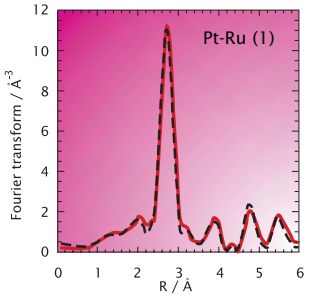
Towards a More Efficient Fuel Cell

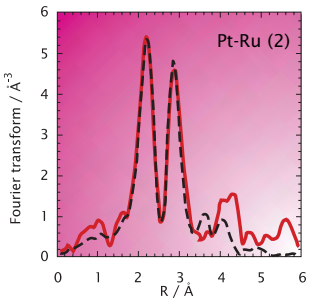
(Revealing Catalyst Metal Alloy Structures)

Fuel cells that use methanol as their source of hydrogen and a platinum based anode catalyst suffer from performance limitations due to catalyst poisoning caused by trace CO from the methanol reforming process.

It is known (empirically) that Pt/Ru alloys perform better in terms of CO tolerance and this is thought to involve sequential reactions at both Pt and Ru sites leading to oxidation of the CO to (non-poisoning) CO2.





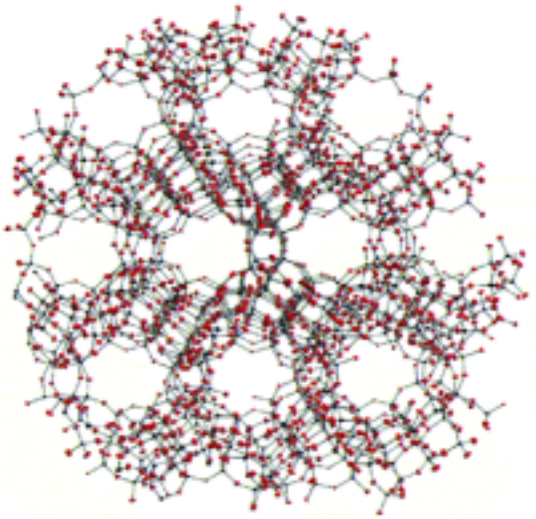


X-ray absorption spectroscopy (XAS) has been carried out on the Pt atoms for two 1:1 Pt/Ru alloys and on Pt alone. The two alloys had been manufactured under different process conditions. Given the proposed mechanism, the objective of the work was to identify the fine structure of the alloy in terms of average near neighbour distances for both Pt-Pt and Pt-Ru atoms.

The results (given in the figure) show that alloy 1 had a Pt absorption spectrum characteristic of Pt itself – indicative of relatively coarse admixing of the two metals at the atomic level (*i.e.* no Ru near neighbours). Alloy 2 however showed two distinct, and fairly equal, peaks – one for Pt-Pt near neighbours and one for Pt-Ru near neighbours.

Further improvements may arise from varying the Pt/Ru ratios and other process conditions now the structural rationalisation for the CO tolerance mechanism has been established.

Zeolite Structures Solved



Zeolites have widespread use in the chemical industry. Large pores in their open framework structure, similar in size to molecules, are linked by a continuous network of channels to form molecular sieves which can act as host sites for catalytic reactions. Based on naturally occurring minerals, zeolites have often been synthesised with no particular end purpose in mind, but now a greater understanding of their structure and function allows their rational design for a specific end use.

Production of zeolites generally leads to powders rather than good quality single crystals. The unique properties of X-rays produced by the synchrotron radiation source used by ***DARTS*** allows much higher resolution powder diffraction data compared with using a normal diffractometer. This is extremely useful when trying to obtain detailed structural information. Additionally, the powder sample may contain microcrystals which can be studied using our single crystal facilities. We are able to obtain structures from much smaller crystals than is possible using conventional diffractometers. The smallest crystal studied so far is 5 µm x 5 µm x 5 µm. The structure shown above is an example of a zeolite structure that was solved using the facilities at Daresbury having previously been unresolvable.

Element specific information can also be obtained from X-ray absorption spectroscopy allowing real time in situ studies of the chemical reactions.

For information on how *DARTS* can help your R&D programme contact us today.