

BCA Spring Meeting

Heriot Watt University 5 April 2000

Controlled environment XRD

This final group session of the BCA 2000 spring meeting at Heriot Watt University rewarded those attending with a number of excellent presentations on techniques using different diffraction experiments in various controlled environments. The last but one timetabled session of the three day event was expertly chaired by Dave Taylor to accommodate six 20 minute talks (of which one delegate remarked "It was good to hear so much pertinent science being discussed....." with reference to the succinct and witty delivery of the invited speakers).

Simon Redfern (Cambridge University) opened the afternoon with a description of novel instrumentation designed to collect X-ray diffraction data from mechanically stressed crystals. Using a three-point bend arrangement with a 120 degree position sensitive detector he described how it would be possible to study ferroelastic materials under conditions where the applied stress is varied. He told the audience that the apparatus was still under construction and showed slides of the computer generated design stage of the manufacturing process. The equipment will be particularly useful in following changes in the elastic shear modulus as materials undergo phase transitions on heating.

Simon rescheduled his talk so that he could catch a waiting taxi and was followed by John Loveday (Edinburgh) with an absorbing talk on neutron diffraction at high pressure with particular emphasis on boron nitride. John informed the audience of the development of the Paris-Edinburgh cell (low mass, large volume) which had now extended the pressure range for neutron diffraction experiments by an order of magnitude up to 30 GPa (300kbar). He also mentioned the high pressure phase behaviour of ammonia monohydrate and explained its importance for formation models of Jupiter's moon Titan and other planetary satellites.

Few of us present will forget without a smile John's last cartoon of two hungry polar bears astride adjoining holes in the Arctic ice. One seen smashing its fist into the water beneath one hole, while the other catches a fish ejected from the other hole with the caption: IF YOU WANT RESULTS FAST, USE HIGH PRESSURE !

Following on and also from Edinburgh University, Andrew Harrison speculated on the claims that the rate or even the course of a reaction may be influenced by microwave heating effects. Although using microwaves has become a routine reaction method in preparative

chemistry "the jury is still out". Andrew produced some outstanding, clear transparencies showing the progress of hydrothermal experiments (involving microwaves as the heating source), which caught the attention of one of my colleagues who is working with more conventional hydrothermal treatments. This was real state of the art stuff and exhilarating too.

To conclude the first part of the afternoon Christopher Frampton (Roche Discovery, Welwyn) warned of the dangers of polymorphic transformations that pharmaceuticals are suspect to in the field, due to variable humidity and temperature. Chris' first slide adequately stressed the importance of storage by depicting a typical pharmacy in a third world country and noting the difference in conditions from off the shelf preparations to the possible phase transitions caused by environmental stressing. His talk concentrated on two compounds creatine monohydrate and Zopiclone and his XRPD patterns clearly illustrated the difference environmental conditions can have on the structure through hydration/dehydration processes. Trials are still under way.

The crisp management of the first part of the afternoon allowed discussion and debate to continue over tea and on return Martin Soar (BP Amoco Chemicals, Sunbury) employing corporate design in his lecture slides (yellow on green) reminded us of the role of in-situ X-ray diffraction in catalyst development. Knowledge of structure and crystallite size is important in understanding activity as well as deactivation of many well-known catalysts. The experiments that Martin described were attempts, albeit sometimes disastrous to the equipment, to study materials under realistic reaction conditions and he managed to chronicle the industrial facility from the mid 1980's to the present day.

And so last but not least John Evans (Durham University) rounded off with a tour de force considering it was the penultimate lecture of the entire three-day conference, he applauded the remaining audience for their "fortitude" in staying the course. John's eloquent discussion of the phase transitions that ZrV_2O_7 undergoes on cooling from a high temperature simple cubic phase via an incommensurately modulated phase to a room temperature $3 \times 3 \times 3$ cubic superstructure were linked to the negative thermal expansion behaviour of the material. Laboratory based X-ray equipment and powder neutron diffraction were the mainstay of John's variable temperature investigation, similar he noted to that used by the other presenters whom we had the pleasure of listening to.

Thanks also go to the organisers of the BCA conference Georgina Rosair and Chick Wilson for arranging a stimulating three days of scientific discussion and exchange of ideas. If this has whet your appetite for more you might be pleased to make a note in your diary of a follow up

workshop on 21st June organised by Dave Taylor and Jeremy Cockcroft at Birkbeck College on non-ambient diffraction techniques.

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