

BCA IG Newsletter

February 2004

EDITORIAL

Welcome to this latest issue of the IG newsletter. There are lots of meeting reports in this issue. I hope you'll find something useful from them. The recent Forum II meeting was a great success. Those of you unable to get to Birkbeck missed two days of excellent presentations backed up by superb local organization. Thanks must go to Jeremy Cockcroft and Dave Taylor. As you can see, there are more meetings coming up. This year's Spring Meeting in Manchester is again of a similar format to last years. The industrial group is responsible for some aspects of the programme. Please have a look at the details in this newsletter and try to come along. As with last year, one-day registrations are available. We are also planning two meetings in June, the first is designed to help newcomers to X-ray diffraction and the second is another in the series of Pharmaceutical SIG's which have proved very popular. Finally, we have our Autumn meeting which this year is on DIY Crystallography. As always at this time I ask for volunteers to join the committee. This year we are again looking for new recruits. Please do think seriously about offering your services. It doesn't take up much time and is something useful to put on your CV! Finally, a big thank you to all those who have written articles for this newsletter. Please note that the BCA web site has a new address, (see opposite).

Phil Holdway

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Forthcoming Events 2004

Registration forms available on the WEB.

BCA SPRING MEETING, UMIST, Manchester,
6th - 8th April 2004 (see p11)
Early Registration deadline 5th March

INTRODUCTION TO POWDER DIFFRACTION
UMIST, Manchester, 10th June 2004

Contact: Judith Shackleton

Pharmaceutical Special Interest Group

24th June 2004,

Venue will be posted on the WEB soon.

Contact: Prof. Chris Frampton

INDUSTRIAL GROUP AUTUMN MEETING

'DIY Crystallography'
Birkbeck College, London
4th November, 2004

Contact: Dr Jeremy Cockcroft.



Charity Registration Number: 284718

World Wide Web addresses:

BCA <http://www.crystallography.org.uk>

IG <http://www.crystallography.org.uk/ig/ig.htm>

****INDUSTRIAL GROUP AGM****

BCA Spring Meeting, UMIST, Manchester
12 noon 7th April 2004.

Further details inside (see page 12)

23rd INTERNATIONAL DURHAM CONFERENCE on X-RAY ANALYSIS

UNIVERSITY OF DURHAM

The 23rd International Durham Conference on X-ray Analysis, organised by PANalytical Limited, was held at the University of Durham from 15th-18th September 2003. There were 4 plenary lectures and over forty lectures concerning the methods and applications of XRD and XRF. These notes are intended to give a flavour of the breadth and depth of the quality of the XRD presentations over the two days of parallel sessions. The opening session dealing with a historical review of XRF and XRD and Recent Developments from PANalytical staff on Monday and the closing session on the Thursday morning on compliance with Environmental Legislation are not covered in these notes.

Stephen Hillier (Macauley Institute) discussed the issue of detection limits in XRPD. He explained that perceptions range from a few weight percent to exact statements found in some industrial methods. Thus, generalisations can be misleading and exact statements for the general method are unsatisfactory. Stephen described how the reference intensity ratio (RIR) method can be used to calculate the lower limits of detection of any phase – whether present in a sample or not. The calculation of detection limits for asbestos in cosmetic talcs was discussed.

Robin Aird (PANalytical) reviewed the hardware and software items that are available from PANalytical. They have positioning stages with movements in the psi, phi, x, y and z directions. Sample alignment and positioning is now possible using a video camera. Several non-ambient stages are available from RT to 1600C with a choice of reducing, oxidising or inert atmospheres. A non-ambient capillary system is available from RT to 700C that reduces preferred orientation. Low temperature cryostat systems are also available. Upgrade options include, Pre-Fix optics, the new X'Celerator detector and new anti-scatter slits for the X'Pert Pro. New software include, X'Pert Quantify and X'Pert Highscore Plus (includes Rietveld analysis).

Judith Shackleton (Manchester Materials Science Centre) described how a portable diffractometer and an AEA MAPS magnetic system are used to measure residual stress in railway rails. In service, the railway line sees high loads at relatively small contact points. The crystal structure starts to break down. The surface is in compression, underneath-tension. Thin slices of track are obtained and longitudinal stress calculated from Hook's Law [Young's Modulus – Stress/Strain] using the $\sin^2\psi$ method. In effect, the crystallographic planes act as an atomic scale strain gauge. The AEA MAPS system can measure to depths of 500 microns and can produce stress distributions as maps.

Richard Morris (Huntsman Surface Sciences UK Ltd) described the Small Angle X-ray Scattering (SAXS) Analysis of Sugar Based Structured Surfactant Systems. This delivery system comprises surfactant, carbohydrate and water. Sugar induced 'Lamellar' sheets are formed with a spacing in excess of 500 angstroms. Richard showed that a 15% sugar solution produced a 315-angstrom bi-layer whereas a 45% sugar solution was not thermally stable. These systems 'flow' yet can suspend solid particles. Richard demonstrated this by passing around several exhibits. He said that we could make similar solutions by mixing washing up liquid (the cheap stuff), sugar and Listerine mouth wash – give it a go!).

Michael Morris (University College, Cork) described a method by which Mesoporous Thin Films (MTF's) are grown on silicon substrates. SEM shows these films to be crack free to thicknesses around 1 micrometer. XRD indicates the presence of pseudo-hexagonally ordered mesoporous systems. Adhesion, film cracking and mechanical strength are properties that need to be controlled. There are about 1,000 million devices on a processor and this is expected to double every eighteen months (Moore's Law). Not every device on a chip is used; every other one is shorted to avoid capacitative 'talk' between adjacent devices.

Kath Clay's (Hexmat Materials Consultancy) presentation covered post measurement

Durham Conference (continued)

transformations of single crystal orientation data (from turbine components). Modified Back Reflection Laue methods are used. Inspection access can be limited as component size and complexity increase. Kath described post measurement orientation data transformations. With three consecutive rotations, you can move from one point of view to another. Stereographic projections are used for the transformations that follow the standard rules for rotation.

Martin Gill (Natural History Museum, London) spoke on the subject of meteorites. A 'meteorite' is material that has fallen to earth whereas a 'meteor' is an incandescent streak of light. Many meteorite types have been discovered, some of which are held at the Natural History Museum. Some are iron rich, others have silicate embedded in the surface, and others are iron-nickel alloys. Martin showed photographs of several impact sites. A meteor crater in Arizona was produced by a 150ft lump of rock that displaced 159 million tonnes of limestone and sandstone, ejected to form a crater 2km across.

Robert Delhez (Delft University of Technology, The Netherlands) spoke about XRD standardisation. Robert explained that standardisation should imply verification, calibration, validation, and sometimes certification - procedures that can prevent diverging results between laboratories. The benefits of standardisation include: an assessment of non-removable systematic instrument errors, quality improvement and an incentive for improving instruments. However, sacrifices need to be made. These include more/longer measurements, more extensive reports and the rejection of non-standard methods.

Chris Staddon (Nottingham University) described X-ray studies of GaMnAs 'Spintronic' materials. For Spintronic materials the ferromagnetic transition temperature has to be greater than room temperature. The magnetic properties of GaMnAs thin films make them suitable materials for Spintronic devices. Chris described how a series of 50nm and 1000nm GaAs thin films with different Mn compositions were grown and analysed with an X'Pert MRD, before and after annealing.

Paul O'Meara (PANalytical) discussed quantification using the Rietveld Method. Paul said

that since the whole pattern is used in the analysis, no crystallographic information is lost. A calculated pattern is compared against observed data and the difference between the two is minimised. Good quality data and structural information is required. The scan needs to be high resolution (5-10 steps across FWHM), high intensity ($>10^4$ counts on the strongest peak) and over a large 2-theta range. Preferred orientation needs to be avoided. The common Rietveld refinement parameters were explained and Paul gave a typical sequence in which to refine them. Rietveld Refinement can be used when preferred orientation is unavoidable, when standards are not available, when site occupancies vary and when peaks overlap.

Alison Burke (Huntsman Tioxide) spoke about the purity of gypsum, CaSO_4 . A time consuming gravimetric method is used, where Ba is substituted for Ca, which drops out of solution as BaSO_4 . This is then weighed. Alison described how Rietveld Analysis is used as a fast alternative method for purity determination. Gypsum samples are scanned from 10 to 100 degrees 2-theta at a rate of 1sec/step (0.05°/step). Comparisons of results from Rietveld (HighScore Plus) and Chemical analysis show a 7% discrepancy. A correction factor is applied.

Overall, the conference comprehensively covered the XRD and XRF techniques. This, combined with good food and a picturesque location, made the meeting a huge success. To those who were unable to attend, you missed a real opportunity to participate in something special.

Mark Farnworth.

Articles Wanted:

Why not put pen to paper and write a short article for our next Newsletter. There are lots of examples to give you some ideas in our Hints & Tips section on the WEB. Don't forget, if you attend a conference, please send in an article about it.

We are also looking to expand the range of Industrial Applications of XRD on our WEB Site.

All we need are a few well-chosen pictures and a few words. How about something on CEMENTS, MINERALS, MUSEUMS, PHOTOGRAPHY, DETERGENTS, PIGMENTS, POLYMERS

DENVER CONFERENCE REPORT

Denver Technical Centre
2003

This was to be my first Denver conference and I had been looking forward to it for some time. My initial concerns over BA flight delays were not realised as I just missed the chaos at Heathrow caused by the August industrial action. My flight was naturally late and overbooked which resulted in an agreeable upgrade to club class. This provided a lot more leg room and a free refreshment/moisturiser pack good enough to use as a 'gift from the US' for my wife! I had sent a 'graduate' student on ahead to ensure her presence at Monday evenings poster presentation. I arrived just in time to mingle at this first poster session but the free drinks (provided at all poster sessions) and time difference ensured my early retirement. Unfortunately this also ensured my 4.00am awakening on Tuesday.

The conference was held at a large conference centre (the 'Denver Tech Centre') that provided the convenience of hotel and conference facilities at same location. However, it was not within walking distance of anywhere, with downtown Denver some miles away.

The principal conference theme focused upon XRD and XRF of art and archaeological objects. Typical parallel conference sessions from Wednesday to Friday were supported by workshops on the previous 2 days. These included discussions of X-ray optics, Rietveld applications, specimen preparation, alignment, and 2-D diffraction.

I took some time on Tuesday morning to talk to several of the commercial exhibitors, (the commercial exhibition was extensive) which included finally meeting the U.S. part of the Bruker operation.

The workshops of particular interest to me concerned intensity distribution measurements using an area detector. Presentations were delivered from both Bruker and Rigaku representatives and there was some debate (not surprising) that centred upon the advantages and disadvantages of wire vs. image plates and curved vs. flat field detectors. Speeds of data collection were also discussed. I had hoped that these, and the other workshops I attended, would have perhaps involved more audience participation and interaction.

The Plenary sessions on Wednesday morning

focused upon the analysis of art and archaeological objects. The four principal speakers, who all gave fascinating and enlightening talks, were from Belgium, Austria, Canada & the U.K. Initially each explained why the analysis of such objects is important for cultural-historical, conservation/restoration, and assessment of authenticity. State-of-the-art XRD and XRF were employed to examine objects as diverse as Frescoes from Jordan, enamels, stone axes, corrosion products and even Napoleon Bonaparte's signature. We were shown how micro-XRD had successfully even been used to map layers through the cross-sections of small paint flakes. The Wednesday afternoon parallel sessions addressed instrumentation, Rietveld applications, high-resolution diffraction and synchrotron applications. I attended the "new instrumentation" session, which consisted of presentations by technical staff from industries, rather than their sales representatives. These included descriptions of quality assurance processes for miniature X-ray tubes (up to 10W from a 3-4cm tube!), new multilayer optics which, when placed in incident and diffracted beams (twin mirrors) are capable of depth profiling, and a new motorised knife edge system for combinatorial screening.

Later on Wednesday afternoon, I took advantage of being in Denver to meet with an old friend and industrial collaborator (another disillusioned U.K. academic who had recently left for the 'greener pastures' of Denver). This also allowed us to tour the bars of downtown Denver. We enjoyed the sunshine, free buses, excellent customer service and the architecture of the railway station. We did not enjoy the major roadworks that seem ubiquitous in Denver. Nicknamed, "T-Rex", these are apparently due to take 5-6 years to complete!

Thursday consisted of more of parallel sessions. Before lunch these included detectors & sources, synchrotron applications, stress analysis and TXRF, and after lunch, optics, stress analysis, industrial applications and quantitative XRF. Thursday was also the day I was due to deliver 2 talks and take a night flight back to the U.K. I chose to attend the synchrotron session in the morning. This was a diverse set of talks included stress-crack analysis studies of magnetic nanostructures, phases of Ni-Si and texture studies using high energy X-rays. For

me, a most intriguing presentation was provided by Professor Stuart Stock (Northwestern University) who showed, using phase radiography, how a newt would regenerate limbs, which surprisingly is not from the top downwards. Unfortunately, my talk in this session seemed to be better than the fire alarm at clearing the lecture room of an audience... surprising as it was the only talk of the conference to show the inside of a working gentleman's "washroom" !

On Thursday afternoon I attended the "industrial applications" session. Presentations included examples of industrial problems investigated by Herbert Goebel (e.g. inclusions within uPVC window frames, glancing angle applications), a description of new funding sources for hydrogen storage systems (e.g. clathrates) and a review of the research facilities at GE. This was delivered by Yan Gao who said, in comparing industry with academia, that "industry sought solutions whereas academia sought problems". Other lectures included descriptions of a novel simultaneous n-IR/XRD system, and an innovative diffractometer designed for very rapid determination of texture in coated tapes (1 pole figure per minute).

I left Denver on Thursday evening feeling tired (it had been a long and very full programme) but convinced that the trip had been worthwhile. Overall the conference was very well organised and had provided an interesting and diverse range of lectures. All of the talks were of high quality. Lasting memories will include the spectacular pancake breakfasts (spectacular in terms of quantity and cost), and the thorough search of my shoes at the airport. I look forward to attending another Denver conference in future when I have saved up enough money.

Keith Rogers

Newsletter Mailing

To keep cost down and to ensure that the newsletter gets to the appropriate people it is essential that we know your correct address. Also if there is a more appropriate contact in your organisation or if you no longer require a copy please let us know by contacting any of the committee officers.

The newsletter is also now posted on our WEB site (<http://bca.cryst.bbk.ac.uk/BCA/IG/index.htm>) If you would like an e-mail notification of the WEB posting rather than a paper copy, then send an e-mail to djtaylor@lineone.net – with the title SUBSCRIBE WEB NEWS

INDUSTRIAL GROUP COMMITTEE 2003 - 2004

Chairman Prof. Christopher S. Frampton

Crystallography Strategic Marketing Manager, Bruker Nonius
B.V. Oostsingel 209 2612 HL Delft

The Netherlands Tel: +31 (0)15 215 25 19 Fax: +31 (0) 15 215 25 00 Mobile: +31 (0) 65 361 87 85

Email: chris.frampton@bruker-nonius.com

Vice Chairman Dr J K Cockcroft

Birkbeck College, Department of Crystallography, Malet Street, London, WC1E 7HX

Tel: 020 7631 6849 Fax: 020 7631 6803

Email: cockcroft@img.cryst.bbk.ac.uk

Secretary/Treasurer Ms J Shackleton

Manchester Materials Science Centre, Grosvenor Street, Manchester M1 7HS

Tel: 0161 200 3581 Fax: 0161 200 3586

Email: judith.shackleton@man.ac.uk

Committee Members

Dr D Beveridge

Ilford Ltd, Town Lane, Mobberley, Knutsford, Cheshire. WA16 7JL

Tel: 01565 650000 Fax: 01565 872734

Email: david.beveridge@ilford.com

Mr M A Farnworth

Pilkington European Technical Centre, Hall Lane, Lathom, Ormskirk, Lancs. L40 5UF

Tel: 01695 54581 Fax: 01695 54506

Email: Mark.Farnworth@pilkington.com

Mr R C E Morris

Huntsman Surface Sciences UK Ltd, ITC Building, Trinity Street, Oldbury, West Midlands. B69 1XB

Tel: 0121 543 3203 Fax:

Email: Richard_Morris@huntsman.com

Dr V B Cooper

Merck Sharp & Dohme Res Labs, Development Laboratories, Hertford Rd, Hoddesdon, Herts. EN11 9BU Tel: 01992 452264

Fax: 01992 460078 Email: brett_cooper@merck.com

Dr N Feeder

Pharmaceutical R&D (049) Pfizer Global R&D, Ramsgate Rd, Sandwich, Kent. CT13 9NJ

Tel: 01304 646386 Fax: 01304 653909

Email: neil_feeder@sandwich.pfizer.com

Co-opted: Dr. S V Norval

ICI plc, Measurement Science Group, Wilton Centre, Wilton, Redcar TS10 4RF

Tel: 01642 435736 Fax: 01642 435777

Email: steve_norval@ici.com

Newsletter Editor: Dr P Holdway

QinetiQ Ltd, Structural Materials Centre, A7 Bldg, Cody Technology Park, Ively Road, Farnborough, Hants. GU14 0LX

Tel: 01252 393117/392159 Mobile: 07754 775832

Fax: 01252 397223 Email: pholdway@QinetiQ.com

ICDD Representative (Ex officio) Mr. D J Taylor

35, Birchley Rd, Billinge, Wigan, Lancs WN5 7QJ

Tel: 01744 893108 Email: djtaylor@lineone.net

BCA IG Forum II Birkbeck College London

13&14th November 2003

Thursday 13th AM

The meeting started with a brief introduction by Jeremy Cockcroft who also thanked Dave Taylor for all his hard work in organising the meeting. Unfortunately, Dave could not be present as his wife Ann was recovering from hospital treatment. We all wish you a speedy recovery Ann.

Opening session – Non-ambient Diffraction + Amorphous Pharmaceuticals.

1. In-situ Diffraction From Materials and Macromolecules Under Microwave Irradiation – Andrew Harrison, University of Edinburgh.

The design and construction of equipment to drive solid-state reactions using microwave radiation and the monitoring thereof using X-ray or neutron scattering was discussed.

Andrew described how fundamental information could be obtained from in-situ studies for e.g. the direct synthesis of magnetite from microwave hydrolysis of Fe²⁺ and Fe³⁺.

The original microwave source was a commercially available microwave oven, which was attacked with a hacksaw and a soldering iron! However, it was soon discovered that these units are unsuitable for sensitive heating control because they always run at full power - only the time of the irradiation can be varied.

This experimentation led to the development of a purpose built instrument in which the microwaves could be "broadcast" by use of an antennae inside a high-pressure reaction bomb.

Issues with thermometry, uneven heating and hotspots were also discussed as were the measurement of cell parameters including lattice expansion.

Future work will include in-vivo studies of biomolecules for e.g. protein "wringing" modes.

2. Non-Ambient Laboratory Powder Diffraction Studies – Synthesis, Kinetics and Phase Transitions – John Evans University of Durham.

In this presentation John described some of the laboratory experiments, which have been made possible by new developments in non-ambient powder diffraction technology including the synthesis of materials, the study of reaction kinetics and phase

transitions.

The equipment used was a Bruker D8 Advance diffractometer combined with a 1500 K furnace, a Cryofurnace (77 – 723 K) and a Cryostat (11 – 300K).

Variable temperature powder diffraction gives lots of new information about the properties of materials for e.g. expansion rates, phase transitions, kinetics, crystallization, chemistry, order-disorder transitions. Structural phase transitions of inorganic oxides, for e.g. framework materials such as ZrW₂O₈, in which the unit cell is found to have a negative coefficient of thermal expansion.

Following the change of width of the diffraction peaks enables the monitoring of domain changes as a function of time.

(This work is available via the RSC website – a "Hot" article).

Synthesis of ZrW₂O₈ has also been performed in the diffractometer.

3. A Review of Methods Used to Quantify Amorphous Content in "Crystals" – Graham Buckton, The School of Pharmacy, University of London.

Graham started his presentation by asking the question – "Is amorphous material important" to which the answer is –

"Yes – very".

Amorphous material is usually thermodynamically unstable, its presence is not normally intentional (e.g. it is introduced during the milling process) and it acts as sites for the absorption of water. Normally present at about the 1-2% level.

How does one study / quantify it? Various techniques available;

- DSC
- XRPD
- Spectroscopy's
- Inverse Phase Gas Chromatography

For example amorphous lactose and amorphous raffinose

Both may contain 1-2% amorphous material, which effect inhalation efficiency and the solubility of drugs for oral delivery.

Amorphous content may be viewed in two different ways;

There may be crystals with a 99% crystalline core and a 1% amorphous surface or 99% of the particles may be crystalline and the remaining 1% is

BCA IG Forum II Birkbeck College London
13&14th November 2003 (continued)

amorphous.

If the product starts at 100% crystalline physical manipulations such as milling will disrupt the surface and introduce an amorphous phase(s?), which will vary with time so the properties of your product are not constant and will vary from batch to batch.

Thursday 13th PM

4. Standardless Phase Quantification of Industrial Coatings – Susan Etok, RMCS Cranfield University.

Susan described the process of plasma spraying of hydroxyapatite onto titanium substrates compared to direct, low temperature electrodeposition for use in prosthetic coatings. It is important to measure the amount of amorphous calcium phosphate in these coatings and this was done using XRD.

5. Understanding Gear Performance with X-ray Diffraction – Brian Shaw, Design Unit, University of Newcastle.

Submarine Gearboxes – makes a change (sorry!)

Statement – “gears fail mainly from fatigue”

Brian described the importance of how gear design and mechanical alignment can be improved from residual stress XRD measurements.

6. Use of Intense Radiation Sources in the Study of Functional Materials – Paul Barnes, Industrial Materials Group, Department of Crystallography, Birkbeck College.

Paul described how neutron and synchrotron sources are enhancing rapid time resolved observation of functional materials using such techniques as XRD.

Three stories were told to enlighten us;

The Brownmillerite story

The Zirconium Hydroxide Story and

The TEDDI story.....

7. Building Your Own Furnace for HT-XRD – Gopinathan Sankar, Davy Faraday Research Laboratory, The Royal Institution of GB.

This paper described some of the in situ cells that have been developed for examining phase transformations in catalytic materials.

8. Using High Temperature X-ray Diffraction with Steel / Aluminium Production: Look Where the Action is! – Stefan Melzer, Corus RD&T, Ceramics

Research Centre, The Netherlands.

This presentation described how production processes can be controlled and improved by following the melting and crystallisation of materials at high temperatures using X-ray diffraction.

9. Inconstant Catalysts – Steve Norval, ICI Measurements Science Group

Amongst other processes, Steve described the importance of XRD in catalyst design noting amongst others the production of ethylene oxide (a major precursor of many surfactants) and the production of margarine from linoleic acid.

Friday 14th November AM

**Industrial Group Award Lecture:
Adventures in Crystallography in the
Gas Turbine Industry – Colin Small,
Rolls Royce PLC, Derby.**

Following an introduction from Judith Shackleton in which Colin's career in XRD at RR was described pictographically using “Haircuts Through the Ages” as it's central theme, Judith presented Colin with his BCA IG award for outstanding contribution to X-ray Analysis which was a molecular model of Diopside (CaMgSiO_3). She also presented him with a model of a Supermarine Spitfire and a pair of the most sought after Vulcan Appreciation Society socks!

After much applause, Colin thanked Judith and the audience and with his normal aplomb got stuck into his presentation. Oh dear, oh dear me. Colin was not up to his normal stratospheric presentational standards – no, this time he surpassed himself! His description and video footage of the Rolls Royce patented goose slicer (more of which later) will long be remembered by the traumatised audience (which included Colin's parents, wife and children). Colin went on to describe the Rolls Royce Trent 900 gas turbine engine, which will eventually be fitted onto the Airbus A380 airliner, which will ferry us all about the planet during our summer hols. (Bye-the-way this engine has a by-pass ratio of 8.1:1 and develops 70,000 lbs of thrust). As aeroplanes fly through the air they ingest any manner of debris; from hailstones through to the major constituent of your Christmas dinner. So, not

BCA IG Forum II Birkbeck College London
13&14th November 2003 (continued)

surprisingly, the testing procedures that these engines have to endure are extremely rigorous. Some of these Colin described, including the hail gun, during which a zillion tons of ice per minute are “shot” at a running engine and the bird injection test, for which there are three standards – the small bird (~2 ½ lb chicken) the medium bird and the large bird, (the afore mentioned goose) of about 8 lbs. Colin pointed out the importance of defrosting the feathery projectiles before shooting them into the engine!

Colin went on to describe the importance of determining the structural integrity of the turbine blades of the engine and how this is monitored using XRD.

The turbine blade is also full of small holes to allow the passage of 70 tons/min of air through the engine and in dusty environments (e.g. deserts) these holes can clog up with dust, which melts (to form Diopside identified using XRD). As the blades are operating at some temperature above their melting point this is not a good thing!

Next was the problem of volcanoes – or more specifically the problems encountered when an aircraft flies through the dust plume of an eruption. The dust particles from an eruption are extremely abrasive and it has been one of Colin’s major tasks over the last few years to understand exactly what goes on inside a gas turbine engine when it swallows umpteen tons of vaporised pumice.

The fan blades of the engine – the ones at the front (and do the bird slicing) - are composed of three sheets of titanium and are hollow. The way in which the crystalline texture of these blades is affected during their manufacture is crucial. Colin went on to describe his use of pole figures, Euler space and orientation distribution functions in his quest for a numerical description of texture of the fan blades that engineers could understand.

Colin concluded his lecture by describing how the turbine rotor shaft is joined using an enormous inertia welding kit. The only way to examine the resulting joint is by neutron diffraction and this was carried out at the ISIS facility at RAL (must have been difficult getting the samples in and out of the hire car!). Another round of applause followed – Colin, X-ray diffraction lectures will never be the same again!

Colin Small – Industrial Group Award 2003.

Photograph of Colin Small presenting his Indutrail Group Award Lecture has been omitted from this PDF version to reduce the file size for download.

The picture can be viewed on the standard Newsletter web pages.

Friday 14th AM Industrial Applications of XRD

1. Microstructural Characterisation of Advanced Materials Using Electron Backscattered Diffraction (EBSD) – Phil Holdway (and H.S. Ubhi) QinetiQ Ltd, Farnborough, Hampshire.

Phil briefly described the theory of EBSD before moving on to some applications. First of all you need an SEM with the sample tilted to around 70° and successful analysis relies on having strain free surfaces. Kikuchi patterns are produced from Bragg diffraction peaks of back scattered electrons from the sample which have an interaction depth on the sample of ~50nm. Data collection is made by scanning over a grid and can be either manual or automatic. For example shape memory alloys – Ti/Ni – the SEM image is used to select a particle for analysis then EDS is used for elemental analysis and EBSD to determine the crystalline properties of the particle – in this way it is possible to obtain complete identification of small particles within an alloy with no need for TEM or XRD. Phil then went on to discuss the analysis of ZrH and ZrH₂ formation on the surface of Zircalloy. And the analysis of grain structure in laser deposited W, a process that enables the formation of complicated shapes that could not be manufactured using conventional machining techniques. (It is much less wasteful to build a structure up rather than start of with a large lump and end up with a pile of swarf).

Phil went on to say that although EBSD is a powerful tool, and can make up to 50 measurements a second, only a very small area is examined when compared with conventional XRD.

INDUSTRIAL CRYSTALLOGRAPHY FORUM II continued

2. XRD and Reflectivity Measurements in the Glass Industry – Mark Farnworth, Pilkington plc.

Mark described how the techniques of XRPD, GAXRD, pole figures and X-ray Reflectivity measurements can be used to examine samples from all stages of glass manufacture, from the crystalline raw materials to the amorphous final product. GAXRD is used to examine thin coatings on the glass surface for e.g. hydrophobic coatings, which disperse water. Refractory materials are examined to determine how much non-crystalline material is present and the amounts of quartz, cristobalite and tridimite. Also, multi-layer stack coatings are examined e.g. Ti ZrO₂ Ag Si. Texture maps can be produced which show the degree of texture in the silver layer of the coatings. The thickness, density, and the top and bottom roughness of each individual layer can be measured.

3. Applications of XRD in the Imaging Industry – David Beveridge, Ilford Ltd.

With the advent of digital imaging technology the decline in sales of silver halide based film emulsions has been swift. So members of the imaging industry have had to adapt just as swiftly and this has had an effect of the type of samples that the X-ray crystallographer can now be expected to examine. David explained that, historically, most of his samples were silver halide film surfaces and included the identification of the composition of processing solutions, now the majority of his samples are from printer-pigmented inks.

Richard Morris

2003 Quantitative Analysis Round Robin

As part of its involvement in the BCA Spring Meeting 2003 at York, the Industrial Group of the BCA organised a Round Robin Quantitative Analysis competition involving an inorganic (mineral) and an organic (pharmaceutical) sample. The mineral sample supplied was a titanium dioxide sample containing a mixture of rutile and anatase, while the pharmaceutical sample contained a mixture of lactose and paracetamol. The Industrial group of the BCA thanks

Huntsman Tioxide for the provision of the mineral sample used in this round robin competition.

All submitted results have been kept anonymous, but a prize of a bottle of champagne was awarded for the quantitative analysis result for the person (or team) who obtained the best (closest) result for each sample.

For the titanium dioxide mineral, a large single sample was sent of the mixed-phase system. For the pharmaceutical, small (pill-sized quantities) samples of both the mixture and individual components were sent.

Results

Titanium Dioxide Mineral

| Laboratory No. | Rutile | Anatase |
|-----------------|--------|---------|
| 1 | 98.90 | 1.10 |
| 2 | 99.15 | 0.85 |
| 3 | 99.30 | 1.50 |
| 4 | 99.16 | 0.84 |
| 5 | - | - |
| 6 | 99.20 | 0.80 |
| 7 | - | - |
| 8 | 99.55 | 0.45 |
| 9 | 98.80 | 1.20 |
| Standard Values | 99.16 | 0.84 |

In a close run thing the winner was laboratory number 4 whose results were spot on!

Pharmaceutical Compound

| Laboratory No. | Lactose | Paracetamol |
|-----------------|---------|-------------|
| 1 | - | - |
| 2 | 83.20 | 16.80 |
| 3 | - | - |
| 4 | 93.20 | 6.80 |
| 5 | 84.20 | 15.80 |
| 6 | 80.50 | 19.50 |
| 7 | 85.20 | 14.80 |
| 8 | - | - |
| 9 | - | - |
| 10 | 86.10 | 13.90 |
| Standard Values | 84.92 | 15.08 |

Laboratory 5 not quite good enough and was beaten by Laboratory 7.

Many thanks to all who took part and hopefully the workshop in York provided the key to improvement for those who were wide of the mark.

BCA IG Forum II Birkbeck College London

13&14th November 2003

Pharmaceutical Parallel Sessions

Over the course of two days three parallel sessions were held covering non-ambient PXRD applications, polymorphism case studies and the analysis of amorphous materials in a series of nine 30-minute lectures.

The non-ambient session was started with an excellent introduction to RELATIVE HUMIDITY CONTROLLED X-RAY DIFFRACTION by Brett Cooper, (MSD), explaining what relative humidity is and how its variation can impact on pharmaceutical ingredients. He described the latest computer controlled system and how it is now possible to mimic the complementary technique of Dynamic Vapour Sorption (DVS). An example of one of these new computer controlled systems, the Bruker/Ansycos Hot Humidity Controlled system giving the ability to stress samples with combinations of high temperature and high relative humidity. Terry Threlfall (University of Southampton) spoke on STRUCTURAL SIMILARITY, DISSIMILARITY AND REPRESENTATION using alklimetal tartars, frusemide and sulphonamides as examples to reveal unexpected relationships between structures.

The use of high pressure was described by Simon Parsons (University of Edinburgh), POLYMORPHISM INDUCED BY HIGH PRESSURE, pushing out the boundaries of polymorph hunting. He described the experimental conditions using a diamond anvil cell, CCD diffractometer and 'fairly modest' pressures (1-15kbar) to search for new forms of formamide, pyridine and glycine. The high-pressure system was also used to study the formation of co-crystals.

Chris Frampton, (Bruker-Nonius) POLYMORPHIC DRUGS SCIENCE, FASHION OR VALUABLE PRODUCTS gave a highly interesting and entertaining talk on the importance of establishing the most stable form of a compound and acquiring the intellectual property rights. He used the examples of Ranitidine, Ritonavir and Paroxetine to illustrate his talk.

The importance of finding the most stable form was emphasised by Dan Cowell, (Pharmorphix Ltd.), POLYMORPHISM STUDIES IN THE PHARMACEUTICAL INDUSTRY. Dan stressed that high throughput screening was only part of the answer and that better focused screening at medium and low throughput could lead to the most stable form with greater understanding.

THERMAL TRANSFORMATIONS-CASE STUDIES by Ron Roberts (AstraZeneca) showed examples of structural changes during heating and cooling using hot-stage X-ray diffraction. Examples of lattice expansion and calculation of thermal expansion coefficients were also shown.

Stephan Watts, STRUCTURAL CHARACTERISATICS OF THE AMPRPHOUS PHASE: A COMPUTER MODELLING APPROACH, (Pfizer Institute for Materials Sciences, University of Cambridge) talked about a new approach to the understanding of amorphous materials using molecular dynamic simulation techniques.

The final two lectures concentrated on novel techniques for

the quantification of the amount of the amorphous phase in materials. Paul Royall, (Kings College, London), APPLICATION OF DYNAMIC MECHANICAL ANALYSIS (DMA) IN THE CHARACTERISATION OF AMORPHOUS POWDER spoke of the application of DMA to powders as opposed to solid samples. Mixtures of amorphous and crystalline lactose were used to establish a linear relationship between the amorphous content and the DMA relaxation strength. The results from a sample of micronised crystalline lactose were more complex and further work is ongoing to understand these results.

Susan Barker, (University of East Anglia), THE USE OF THERMALLY STIMULATED CURRENT SPECTROSCOPY (TSC) IN THE STUDY OF AMORPHOUS AND POLYMORPHIC MATERIALS, described TSC as an electrical technique whereby dipolar movement and relaxation is measured under the influence of varying thermal and electrical stresses. The various modes of operation of TSC were described in relationship to caffeine and indomethacin.

In all, a very interesting session for all those interested in the characterisation of pharmaceutical solids.

Friday 14th pm Joint Session

The final session of the forum was devoted to the theme of X-ray Diffraction: Past, Present, and Future. With the meeting being held in the Clore management centre in Torrington Square, Birkbeck College, then it was highly appropriate that Alan Mackay should open the session with a lecture on the past. — the old buildings of the Crystallography Department as founded by J.D.Bernal used to stand on the adjacent site. Amongst the many photos of old equipment, he showed a picture of Bernal's X-ray rotation camera — and then contrasted it with the "big science" now done at the ESRF. Probably though the biggest changes have been in computing as shown by the photo of an early computer at Birkbeck College from the 1950s laboratory of Donald Booth. The second talk was by Judith Shackleton on present X-ray instrumentation, a difficult talk to give without advertising the advantages of one manufacture over another. After working her way through the maze of primary and secondary optics, monochromators and mirrors, area and solid-state detectors, she arrived at the "Portable Stress Diffractometer" — a most remarkable piece of kit capable of measuring stresses in very large objects indeed. One wondered what the response would be at Birkbeck College to its lack of radiation shielding! The Industrial Group committee decided that the final talk of the forum should be an Alun Bowen lecture given the importance of future instrumentation for industrial crystallography. The talk on the new UK synchrotron source was given appropriately by Alexander Korsunsky, who described the features of the beamline JEEP proposed for DIAMOND. The talk ended with an aerial photo of the DIAMOND site showing that construction was now seriously underway and the "future" lay just ahead.

BCA Spring Meeting 2004.

UMIST, Manchester, 6th – 8th April

There is considerable overlap with the Groups at this meeting and it is important that you review the full programme on the BCA meeting pages. We concentrate here on items of particular interest to the Industrial Group.

NOTE: Day registration is only **£65** if you book early and 3 days fully inclusive of registration, meals, two nights basic accommodation and conference dinner is only **£228.70**. Fully inclusive rates are available of less than **£150** for students and **£165** other concessions.

Industrial Group Highlights

The IG has put together a comprehensive programme. You have the opportunity to get some real value for money training from world class experts for yourself, a trainee or student. There is something to keep you occupied over the full three days including a comprehensive exhibition featuring all the major suppliers, a real chance to update your product knowledge in a fast changing market place. There will be an Exhibition Open Evening on Monday 5th April 19:30 - 21:30 after the Young Crystallographer Session and Lonsdale Lecture - see BCA Web pages for further details.

Instrument Calibration, How to be a star!

Chair: Jeremy Cockcroft

Session 1: Wednesday 08:30 - 10:00

08:30 - The aim of this introductory session is to explain the criteria of a "star" (*) quality pattern in the Powder Diffraction File (PDF). Describe the procedure and requirements for submitting new "star" patterns to the database.

We will also look at the checks needed to ensure that your equipment gives "star" quality data and the standards and tools that can help you. *Dave Taylor et al.*

09:30 - Aspects of calibration in SAXS, *Manfred Kriechbaum, Institute of Biophysics and X-Ray Structure Research, Austrian Academy of Sciences*

Session 2: Wednesday 10:30 - 12:00

10:30 - European Standards, *Steve Norval, ICI plc.*

11:00 - Flat Plate & Capillary Systems, *Martin Vickers, Birkbeck College.*

11:20 - Stress Instruments, *Judith Shackleton, University of Manchester.*

11:40 - Calibration for Silver Halides, *David Beveridge, ILFORD Imaging UK Ltd.*

12:00 - Lunch

Parallel Session: "Catalysts in Industry"

Wednesday 7 April 15:30 - 16:30

Chair: Steve Norval, ICI plc.

15:30 – Title To Be Announced. *Tim Hyde, Johnson Matthey.*

15:50 – Title To Be Announced. *Steve Norval, ICI plc.*

16:10 – Title To Be Announced. *Speaker from the University of Glasgow.*

Small Angle Scattering Session 1.

Wednesday 7 April 13:30 - 15:00

Chair: Richard Morris

13:30 – SAXS Interpretation of Deflocculated Vesicles, *Richard Clapperton, Huntsman Surface Sciences.*

13:50 – X-Ray time-resolved SAXS measurements, *Manfred Kriechbaum, Institute of Biophysics, Austrian Academy of Sciences.*

14:10 – Some General Aspects of SAXS (provisional title), *Mary Vickers, Cambridge University.*

14:30 – In Situ Diffraction Studies of Microwave-Driven Processes in Materials, Chemistry and Biology, *Andrew Harrison, Edinburgh University.*

Small Angle Scattering Session 2.

Thursday 8th April 10:30 - 12:00

10:30 - Title To Be Announced. *Dmitri Svergun Group Leader, European Molecular Biology Laboratory, Hamburg Outstation.*

11:00 - "He Scrubs Up Well, Doesn't He?" - New Aspects of Expanded Lamellar Surfactants - Sugar Based Exfoliates. *Richard Morris Huntsman Surface Sciences*

11:20 - Title To Be Announced. *Gordon Cressey and/or Colleague Natural History Museum*

11:40 - SANS - Practicalities and Prospects. *Dr. Richard Heenan ISIS Facility R3 1-21 RAL*

Small Angle Scattering Session 3.

Thursday 8th April 13:00 - 14:30

Chair: Mary Vickers

13:00 - Scattering From Segmented Co-polyurethanes - A Reinterpretation of SAXS Data. *Dr. Peter Laity, Cambridge University*

13:30 - X-ray Reflectivity Measurements in the Glass Industry. *Mark Farnworth, Pilkington plc*

14:00 - Title To Be Announced. *Speaker to be confirmed*

| Spring Meeting 2004 Industrial Highlights | | | |
|--|--|--|---|
| | Tuesday 6 April | Wednesday 7 April | Thursday 8 April |
| AM | Young Crystallographers cont. Registration | Instrument Calibration How to be a star! | Plenary Session: Use of International Tables |
| Coffee & Exhibition | | | |
| AM | Plenary Session | Instrument Calibration Continued 12:00 IG AGM | Small Angle Scattering 2. |
| Lunch & Exhibition | | | |
| PM | Extraframework Species in Zeolite Y at Non-Ambient Conditions. <i>Jim Kaduk, BP Chemicals, USA</i> | Small Angle Scattering 1. | Small Angle Scattering 3. |
| Tea & Exhibition | | | |
| PM | Bragg Lecture <i>Prof J L Finney</i> Diamond SIG | Catalysts in Industry | |
| | 18:00 Dinner | 17:30 Dorothy Hodgkin Prize Lecture <i>Prof G M Sheldrick FRS</i> | |
| Evening | 19:00 Posters Exhibitors Wine Reception | 19:30 Conference Dinner | |

Industrial Group Posters

Posters are invited for display at the Spring Meeting. As an extra incentive to your participation, in addition to the acclaim that your poster will no doubt bring, the Industrial Group are offering a magnificent prize of £50 for best poster.

Some guidelines follow for what we would prefer to see in our posters and our adjudicators will work from these.

Posters are encouraged that:

- are relevant to industry (including some background and value of the work to industry)
- have clear aims, results and conclusions
- concentrate on telling the story, rather than fine detail
- are not an advertisement for a commercial product

There will be an opportunity to give a brief oral presentation of the content of each poster.

For more information, contact:

Secretary/Treasurer

Industrial Group AGM

The 21st ANNUAL GENERAL MEETING of the Industrial Group will be held at UMIST at 12:00 on 7th April 2004.

Nominations are sought for **Chair, Vice-Chair** and **two** committee members to serve for three years from April 2004.

Nominations, which shall be proposed by not less than two members of the Group and shall be accompanied by the written consent of the nominee, shall be sent to reach the Honorary Secretary of the Group not later than seven days before the Annual General Meeting.

Contact the Secretary/Treasurer