BCA IG Newsletter

August 2006

EDITORIAL

Welcome to the BCA Industrial Group Newsletter and my first as its Newsletter Editor.
You will find in its pages plenty of information concerning forthcoming meetings and reports of meetings held this year.
Planning is now well advanced for next years BCA Spring meeting at the University of Cantebury. 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. I have included some initial information concerning the content of the XRD and XRF sessions at the meeting. Check out both the BCA web pages and the Industrial Group web pages for more information.

The Newsletter also contains a comprehensive set of reports from the BCA Spring Meeting 2006 at Lancaster University. Thanks goes to all those who took time out from their busy work and home(!) lives to put together the reports.

Congratulations to Paul Fewster (Panalytical), the winner of the Industrial Group award at the Spring Meeting, for his contributions to industrial crystallography and the BCA. (see page 2).

Inside This Issue

Forthcoming Events 2006/2007

9th November 2006 **Autumn Meeting**
Pilkington plc, Lathom, Lancs
Call for papers! more details on the web

17th to 19 April **Spring Meeting 2007**
University of Canterbury
Includes a full XRF programme and exhibition

Log these dates in your diary NOW!

Provisional Diary Dates for the longer term:

14th May 2008
Joint BCA/RSC XRF meeting.

XRF Newsletter 3 published electronically in August 2006. View a copy on the web.

Industrial Group

BCA

Charity Registration Number: 284718

World Wide Web addresses:
BCA http://www.crystallography.org.uk
IG http://www.crystallography.org.uk/ig/ig.htm
Tip Google BCA IG (with a space) to find us!

THANK YOU

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EDITORIAL *Continued*

For you XRD people with an interest in XRF page 11 gives you some information about activities in this area. However, get yourselves onto the XRF mailing list (see below) to be kept even more informed.

Planning is now underway for the Autumn meeting at the Pilkington European Technical Centre, Lancashire (see page 4). Here's your chance to not only hear some good presentations on Industrial Crystallography but also to take a tour of the Exhibition Centre which highlights the many applications of glass and coated glass products. See you there.

Mark Farnworth
Newsletter Editor

Industrial Group E-mail Mailing lists – Online registration.

We now maintain separate lists for XRF and XRD mailings so please register for BOTH if you want to be kept totally in the picture. The IG sends about six E-mail notices each year to anyone interested (You don't even need to be a BCA member!). These inform of Newsletter postings and the various meetings we organise each year. You can now register for our E-mail lists online - follow the link from the IG home page. There is an opportunity to be removed from the list with each mailing.

Sudoku: Solution to David Beveridge's puzzle from the last issue.

1	4	7	2	5	8	3	6	9
2	5	8	3	6	9	1	4	7
3	6	9	1	4	7	2	5	8
4	7	1	5	8	2	6	9	3
5	8	2	6	9	3	4	7	1
6	9	3	4	7	1	5	8	2
7	1	4	8	2	5	9	3	6
8	2	5	9	3	6	7	1	4
9	3	6	7	1	4	8	2	5

Sponsor this Newsletter!

Sponsorship of the distribution of around 400 copies of our next Newsletter is sought. Sponsorship offers good value and covers the full cost of distribution (approximately £200) – *contact the web editor for full details*.

Industrial Group Award

2006 Award to Prof. Paul Fewster (PANalytical)

Paul (right) receives his award from Jeremy K Cockcroft the IG chair.

Professor Paul Fewster is widely acknowledged as one of the leading experts on X-ray diffraction world-wide. He has made major contributions to the development of both the apparatus and the analysis of the results and has used this expertise to contribute to the development of a wide variety of electronic and photonic devices.

Over the years, first at Philips and later as an independent scientist, Paul has influenced the development of many semiconductor devices. This began with his early studies of III-V compound semiconductors such as AlGaAs, where he did much pioneering work on the structural quality of multi-quantum well structures, which now form the basis for all solid state lasers. He extended this work to include the much wider family of III-V, II-VI and silicon based structures, most recently including spintronic materials and quantum dots.

Paul has also made major contributions to the work of both the Institute of Physics and the British Crystallographic Association being active in both bodies for many years. His expertise has been acknowledged by his appointment as a visiting Professor of Physics at Imperial College.

Paul has made an *enormous* contribution to the BCA in several areas: he was a hard working member of the PCG committee for many years (1983-1996) serving in many rôles during this period, then after a short break he agreed to serve on the Industrial Group committee. In addition, during this latter period he was elected Vice-President of the British Crystallographic Association for 2001-2004, which at that time involved organising the programme for the BCA Spring Meetings. In summary, Paul has made an invaluable contribution to the application of X-ray diffraction to a variety of scientific problems and major commercial applications.

2007 Spring Meeting University of Canterbury
17-19 April 2007 Industrial Group XRD Highlights

	Tuesday - 17 April	Wednesday - 18 April	Thursday - 19 April
AM	Registration & Exhibition	Keynote.	Teaching Keynote
Coffee & Exhibition			
AM	Lonsdale Lecture	XRF / XRD Joint session on thin films. Bragg Lecture	One Hundred and One Ways to Prepare an XRPD Sample.
Lunch & Exhibition			
PM	Co-crystals of Pharmaceutical Materials	13:00 IG AGM IG Keynote Prof. R L Snyder.	A Standardless Future for Quantitative XRPD?
Tea & Exhibition			
PM	Exhibitors talks	Diffraction fromSurfaces and Two Dimensional Crystallography. 17:00 BCA AGM	
		17:45 Hodgkin Lecture	
Evening	18:30 Posters & Exhibition - Buffet & Wine Reception	19:30 Conference Dinner	

Individual Session Details Follow

Co-crystals of Pharmaceutical Materials

Tuesday 17th April, 15:15 - 16:45
Chairs:
Anne Kavanagh, *AstraZeneca, Macclesfield.*
Roy Copley, *GSK, Harlow.*

XRF / XRD Joint session on thin films.

Wednesday 18th April 2007 10:15 - 12:00
Chairs:
Dave Taylor, *ICDD*.
Chris Staddon, *University of Nottingham*.
This joint session is designed to give delegates an insight into what can be achieved by using XRF and XRD to investigate thin films and coatings. We are planning to start the session with an overview of what can be achieved and follow with presentations on specific applications of the techniques.

IG Keynote Lecture,

Wednesday 18th April 2007, 14:15 - 15:00
Professor R. L. Snyder, Georgia Institute of Technology

Diffraction from Surfaces and Two Dimensional Crystallography.

Wednesday 18th April 2007 15:30 - 17:00
Chairs:
Judith Shackleton, *University of Manchester*.
Richard Morris, *Morris Analytical X-ray*.
During this session we hope to cover; Glancing Incidence Scattering and Diffraction; New advances and applications of 2D detectors - for example diffraction from fibres, composites, carbon fibres etc.; Spatial mapping of composition and residual stress. We also hope to include small angle scattering, SAXS looking at "soft" materials and possibly non-ambient applications. Just about any thing which can be done with a diffractometer in space and time.

One Hundred and One Ways to Prepare an XRPD Sample.

Thursday 19th April 2007 10:15 - 12:15
Chairs:
Jeremy Karl Cockcroft, *UCL.*
Martin Gill, *Natural History Museum.*
NB. Session to include the IG prize talk by a Young Crystallographer.

A Standardless Future for Quantitative XRPD?

Thursday 19th April 2007 13:30 - 15:00
Chair:
Steve Norval, *ICI, Wilton.*
Standardless XRPD now appears viable with methods based on crystal structure data and others on the Powder Diffraction File. Are these techniques really ready for us to use? Is quantitative or semiquantitative analysis becoming routine without the hassle of the old standards based methods? Is there still a place for analysis with standards? These are questions with relevance to many XRPD labs and contributions are invited towards establishing the state of the art in real laboratories.

Industrial Group and XRF Posters

Posters are invited for display at the Spring Meeting - magnificent prize of £50 and a bottle of Champagne 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

For more information, contact:
Secretary/Treasurer (see back page)

Industrial Group AGM

The 24th ANNUAL GENERAL MEETING of the Industrial Group will be held at Canterbury at 13:00 on 18thApril 2007. Nominations will be sought to fill vacancies for **Chair and three** committee members to serve for three years from April 2007. The committee works hard in the background, planning the I.G sessions at the BCA Spring meeting and planning the Autumn and Special Interest Group meetings. Do you have IT or organisational skills that you could put to work for the Industrial group?

Autumn Meeting 9th November 2006
Pilkington European Technical Centre, Lathom, Lancashire.

Impact of Crystallography in an Industrial Environment 10:00 – 4.30pm

CALL FOR PAPERS on the theme *"Impact of Crystallography in an Industrial Environment"* - offers of talks should be sent to the local organisor Mark Farnworth. Some ideas for contributions based on the theme are listed below.

Process Monitoring:
Analysis/quantification of airborne dusts, waste and sludges.
Analysis/quantification of Raw materials
Monitoring of plant and equipment for corrosion and wear (strain?)

Product Characterisation:
In what ways can XRPD data characterize products to determine if they are faulty / out of specification?
How can XRPD data (e,g. identification, texture data, strain data)link with other analytical data to solve industrial problems
High throughput XRPD - challenges

Speakers include: Chris Gilmore, Ian Ferguson, Mark Farnworth and Ivan Parkin.
Finding the venue, Postcode L40 5UF: A minibus will leave Wigan North Western railway station at 9.30am. Contact Mark Farnworth at least 3 weeks in advance if you want to use this facility

During the lunch break there will be an option to take a tour around the on-site Exhibition Centre which highlights the uses of glass and coated glass products in the Building and Automotive markets.
Meeting Registration will be online from the IG meeting web page. The web will be updated with the latest programme information so please check regularly for details.

Reports - Spring Meeting 2006, Lancaster 4th to 6th April 2006

Phase Identification Modular Workshop 4th April 2006

Photograph of some of the workshop delegates

The modular structure and informal approach of the workshop allowed conference delegates to come to the sessions that best met their requirements. Between ~10 and ~30 people attended each session. There was plenty of opportunity to question the experts both during and after the presentations.

David Rendle, a volunteer editor with ICDD with years of experience in the metropolitan police forensic science lab, led a session on the History and Structure of the ICDD Powder Diffraction File, from its inception very early in the history of XRD, to the present. The first set of hand-written cards was produced in 1941, with Set 3 in 1949 comprising ~2500 patterns. Today, nearly half a million patterns are available on electronic media, covering both experimental and calculated patterns. Information is gathered from all over the world and carefully reviewed before publication.

The session on Phase Identification was led by David Taylor, currently ICDD volunteer treasurer and BCA IG web editor. Considerations for good measurements were discussed (such as scan parameters and instrument settings) and an understanding of the instrumental errors and effects different properties of the sample can have on the results, e.g. if it is textured or stressed. Sample preparation methods to minimise induced texture and stress were mentioned, and the usefulness of certified NIST Standard Reference Materials for characterising the instrument and providing an internal reference in a scan.

Building on David's introduction of the main methods for identifying phases (alphabetic searches, Hanawalt, Fink and Long8) John Faber of ICDD gave more detail in his Advanced session, with explanation and discussion regarding quantification of phases within a specimen. The importance of applying all knowledge about a sample in order to obtain the most robust conclusions was brought out, e.g. any information from other measurement techniques, its physical properties (e.g. colour, density) and its history.

The use of reference scans, concentration curves and internal standards are important for the most precise analyses, together with thorough understanding of instrumental and sample effects on the data, and at what level these need to be taken into account. Gather good evidence and argument in order to be sure of your conclusions. As ever, GIGO applies.

The last workshop was a hands-on computer session with PDF4+, DDView+ and Sleve+ software from ICDD. Many thanks to the IT staff at Lancaster who prepared PCs in a suite so that each delegate could use the software. This was valuable experience, especially with expert guidance and the opportunity to ask questions throughout. We were working through standard examples which are usually used with a reduced PDF set. As such, some of the matches came out differently from the text-book answers - different card numbers for similar material. I was about to be concerned about this, but different cards for the same type of material can reflect for example the effects of different methods of sample preparation or the data quality, and do not necessarily mean that I've got the answer "wrong"!

Tamzin Lafford
Bede X-ray Metrology.

Neutron & Synchrotron Opportunities for Industrial User. 5th April 2006

Speakers - Left to Right: Alan Hewat, Jeremy Cockcroft, Andrew Jupe.

Reports - Spring Meeting 2006 – contd.

Alan Hewat (ILL)[*] described the *High Flux Neutron Diffractometers at ILL Grenoble.* The ILL, a CCLRC laboratory, is open to all UK scientists provided that the work they do is published. The following instruments at the ILL are about to come on line again after an eight month period of refurbishment. A call is presently out for new research proposals before an August 2006 deadline. The following equipment is available on the high flux beam lines from the ILL reactor: SALSA [(1)], a neutron strain scanner capable of determining strains in industrial components weighing up to 0.5 tonne. Work on this is supported by the FaME38 industrial support laboratory [(2)].

The super-D2B detector [(3)] which yields very high resolution neutron powder patterns in much less than an hour per temperature step.The D20 high flux powder diffractometer [(4)] which can collect complete diffraction patterns at 1-10 Hz thus enabling chemical kinetic studies to be carried out. Its rate of data acquisition is comparable with a synchrotron. The D20 can be used for example to examine ordering in metal oxides, and the detailed structure of fullerene based structures. Explosive reactions such as that between Ti, Si and C can also be studied at 300msec intervals. Its furnace can reach 2200 degrees Celsius, whilst 100kbar pressures can be applied during experiments.

The new position sensitive detector on D19 [(5)] greatly accelerates data collection on organic fibres and protein single crystals. VIVALDI [(6)], a neutron image plate Laue camera can collect complete diffraction patterns from small single crystals in 30 minutes. DRACULA [(7)] is an order of magnitude faster than D20, and can be used similarly. CYCLOPS [(8)] (CYlindrical Ccd Laue Optics Photo-Scintillator will provide real time diffraction patterns covering 70% of 4pi in sub-second read-out time. All these instruments are designed to optimise the use of a continuous neutron source, and to complement new machines on pulsed neutron sources e.g. IBIS, which offer different advantages.

[*] *Diffraction Group, Institut Laue-Langevin, B.P. 156X Grenoble Cedax 9, FRANCE.*

See the web version of this for the useful links Alan made available.

Andrew Jupe (Georgia Institute of Technology, USA) spoke on *In situ synchrotron diffraction for studying oil well cement hydration at elevated temperatures and pressures in real time.* He described the advantages of X-ray diffraction using a synchrotron, and how he had applied them to the hydration of Portland cements at both elevated temperatures and pressures. The experimental conditions used were based on the environments encountered in an oil well where Portland cement is used as a grout between the bore of the oil well and its steel liner. The cement mix is forced down the well using pressures of several kilobars whilst the ambient geothermal) temperature can reach 250 degrees Celsius. Five unambiguously identified Bragg reflections at high d-spacings were used to monitor the evolution of the five phases which occur in the final product cement. These phases crystallise from the calcium hydroxide and amorphous calcium silicate hydrate phases which are formed initially. It was shown that the presence of tartaric acid appears to influence the formation of aluminosilicate gels in the mixture; whilst pressure had a strong influence on the beneficial formation of tobermorayite which is formed when silica flour and zeolite are added to the initial mix.

Stephen Thompson (Diamond Light Source Ltd) gave an *Update on powder diffraction on Diamond.* This facility is planned to be operational in August 2008. The high flux available will enable high user throughput as well as very high rates of data acquisition. Data will be collected using X-rays with wavelengths between 0.4 and 2.5 Angstroms. The user hutch will be 9m by 4m in size, with user and preparation laboratories on hand. A double bounce X-ray mirror will be used, with Si, Rh and Pt strips to avoid the presence of harmonics in the beam. The size of the X-ray beam will be 15 X 2 mm at 5kV and 6 X 1 mm at 20kV. Particular design efforts have been made to provide a user friendly facility, which will avoid the need for the user to optimise the experimental conditions as well as minimising the time spend on user training.

Ian Ferguson
Retired (UKAEA)

Reports - Spring Meeting 2006 – contd.

Crystallisation and Polymorphism of Pharmaceuticals

5th April 2006

Speakers - Left to Right: Caroline Day, Anne Kavanagh, Terry Threlfall, Sally Price, Roy Copley, Roger Davey, Ulrich Griesser.

The **Alun Bowen Lecture** was given by **Ulrich Griesser (University of Innsbruck)**, who discussed polymorphism in drug development (more than half the drugs in the European Pharmacopia) are listed as forming polymorphs or hydrates/solvates). His lecture illustrated how thermal microscopy can be used to understand this phenomenon and to determine the relationships between different forms. For instance, solid-solid transformations can be readily detected in the hot-stage microscope, but difficult to detect by DSC. In addition, crystallisations on the hot-stage microscope can be used to generate polymorphs which might be difficult or even impossible to achieve via crystallisation: this can be done by sublimation from a heated microscope slide onto the cover slip and by crystallisation from the melt followed by thermal cycling.

The session on ***Crystallisation and Polymorphism of Pharmaceuticals*** opened with **Roger Davey (University of Manchester)**, who considered nucleation from solution, and how it affects the outcome of crystallisation. There is a growing belief (based on computer simulation and some experimental evidence) that on nucleation an amorphous short-lived entity is formed. In another case (tetrolic acid) there is a direct relationship between self-assembly in solution and the H-bonding motifs in the resulting crystals. He considered mandelic acid, and the formation of the racemic compound versus the formation of a conglomerate. It was not possible to crystallise the conglomerate in preference to the compound from a racemic solution (even when seeded with the conglomerate). But if the solution contained an enantiomeric excess, and suitable additives were used, then it was possible to achieve enantiomeric enrichment in the crystals.

Sally Price (UCL) talked about progress and problems in crystal structure prediction. She described success in the prediction of the crystal structure of planar molecules, such as 3-oxauracil. Difficulties arise when predicted energy landscapes have many polymorphs with very close energy minima, and also noted that kinetic factors might prevent nucleation or growth of some structures. A further difficulty arises from the fact that many compounds of practical interest, such as pharmaceuticals, are floppy and hence may adopt different conformations in different solid forms. Combining crystal structure prediction with experimental work was used in an automated polymorph screen of carbamazepine: the six known forms were found, along with three solvates. However, a polymorph with a chain motif, that was predicted, was not found experimentally. It was found that the polymorphic form was dependant on the crystallisation protocol rather than on the solvent.

Terry Threlfall (University of Southampton) gave a personal overview of crystallisation and polymorphism studies. He advised that practical choices, such as container fabric, storage time, separation and drying regimes could all affect the polymorph obtained, as well as the more obvious variables. The chances of obtaining a novel form are maximised when crystallisation occurs in confined spaces, such as a capillary, hanging drop or emulsion, since the mother liquor is rapidly depleted once crystallisation occurs. His cautionary tales included how a previously unknown but thermodynamically stable form of the drug Ritanovir, appeared once the drug was on the market. The stable form was templated by the presence of an impurity, and its appearance required a total reformulation of the medicine to accommodate it. Terry left us with one last

thought: that in isolation, the least useful piece of information you can have about a polymorph is its crystal structure.

The last talk was given **by Caroline Day, of GSK,** on polymorph screening by automated techniques. Pharmaceutical companies need to make a suitable choice of solid form (i.e. the right salt and polymorph) fairly early in the development of a drug, so as to minimise the chances of nasty surprises and expensive changes later on. However, in early development the quantity of drug available is quite low, and as development progresses, the proportion of drug candidates that fail (for instance, due to toxicity) is very high. Caroline described how early development screens use automated crystallisation and analysis where possible; experimental variables include crystallisation method, solvent, temperature, supersaturation and slurries to identify the stable form. In addition, the design of the crystallising vessel, including the choice of material, is very important in firstly maximising the chances of obtaining a solid, and then being able to analyse that solid *in-situ*, by XRPD and/or Raman.

Anne Kavanagh
Astrazeneca.

Crystal Structure and Growth at the Nano-Scale

Speakers - Left to Right: Moti Lal, Richard Morris, Kevin Roberts, Klimentina Pencheva and Peter Laggner.

How Crystals Are Born: Novel Insight from Small-Angle Scattering.

Peter Laggner, Manfred Kriechbaum and Heinz Amenitsch *Institute of Biophysics and X-Ray Structure Research, Austrian Academy of Sciences A-8042 Graz, Austria*

In principle, the events that eventually lead to nucleation and growth of crystals from solutions or amorphous solids must involve density fluctuations at the nano-scale. This is the natural domain of small-angle scattering. The most general, direct observable is the specific inner surface, which is related to size and volume fraction of the fluctuations. The question is whether present X-ray techniques are sufficiently sensitive to detect the relevant pre-nucleation events. The lifetimes of these fluctuations are generally in the sub-microsecond range and their abundance at any instant, and the density differences between amorphous and nascent crystalline state, are extremely small.

Technical innovations in small-and wide-angle scattering, mainly concerning the brilliance of X-ray sources (synchrotrons), refined X-ray optics with low background, and efficient detectors have opened this field for new experiments. Basically, three types of experiments can be distinguished: (a) observation of the equilibrium fluctuations at p, t, c - conditions below the thermodynamic transition boundary, (b) steady-state, real-time observation of fluctuations during slow-scan conditions, and (c) rapid jump-relaxation experiments. While (a) and (b) can be performed with advanced laboratory X-ray equipment, (c) essentially requires the brilliance of synchrotron facilities.

Experiments were presented on crystallizing systems from solution, from amorphous solids, and from liquid-crystalline phase transitions. *Read more on the experiments on the web!*

Solution Phase Nucleation: Cluster Size and Shape and its Correlation with Kinetics and Polymorph Selection.

Kevin Roberts University of Leeds.

Kevin introduced the importance of surface crystallography in nano-crystals. This is not usually considered, as one is usually looking at large crystals where surfaces are not important. It is essential to be able to predict crystal morphology and to do this Kevin described a software suite - POLYPACK that can define a polyhedron and pack it with molecules. With such a model one can predict crystallinity, RDF, geometric scaling and calculate equivalent diameters surface area and volume.

Reports - Spring Meeting 2006 – contd.

Kevin presented examples of aspirin, glutamic acid - a and ß forms and d-mannitol and described the dependency of stability on size and nucleation rates vs. cluster sizes and asked the question; which polymorph do we want linked to nucleation? To decide this his team modelled small crystalline structures using XRD and the Polypack program.

Metal Nanocrystallites in Supercritical Fluids The Solvation Process and Its Impact on the Nanostructure
M Lal University of Liverpool
In investigating the effects of solvation and passivation on the equilibrium structure of metal nanoclusters dissolved in supercritical fluids, we have performed Molecular Dynamics simulations of bare and passivated 38-atom gold nanoparticle in ethane at several isotherms in the supercritical regime. The bare nanoparticle is found to be appreciably solvated with the solvation layer comprised of two regions: the inner region, located next to the cluster surface, making a dominant contribution to the degree of solvation, and the outer region making a relatively small contribution. The solvation gives rise to large distortion of the in-vacuum, minimum-energy, truncated octahedral structure of the particle, due essentially to the strong the solvent/metal atom interactions present in the solvated core.
See the web report for more detail and pictures.

Quantifying Solubility Enhancement due to Particle Size Reduction and Crystal Habit Modification: Case Study of Acetyl Salicylic Acid. **Klimentina Pencheva,** Univ. of Leeds.
The poor solubility potential drug molecules is one of the major problems facing pharmaceutical scientists. It is well known, however, that the solubility of crystalline materials is enhanced with the reduction of particle size to sub micron levels. Surface molecular modelling was used to calculate the specific surface energies of different (hkl) faces of aspirin (acetyl salicylic acid) as a difference of the surface vacuum energy and surface solvent binding energy. The solvent binding energies were found using systematic search methods combined with solvent molecule docking on crystal surfaces. Using the modelled interfacial energies, the solubility enhancement as a function of the reduction of particle size was calculated as a function of growth morphologies for aspirin and solvent composition.
View the full session report on the web.

Richard Morris,
Morris Analytical X-ray Ltd

Powder Diffraction in Industry

Speakers - Left to Right: Martin Gill, David Beveridge, Judith Shackleton, Chris Staddon, Mary Vickers, Andrew Hodge, Paul Fewster.

The citation for the **Industrial Group award to Paul Fewster** is reported with photographs on page 2.

The session started on time, after a short lunch. **Judith Shackleton** (chair) introduced **Jeremy Cockroft** who gave a brief introduction and awarded the Industrial Group prize to **Professor Paul Fewster**, in recognition for his sustained contribution to industrial crystallography including crystallographic and diffraction work in industry of all kinds.

The award lecture was titled *It's all in the detail*. Paul initially talked about his previous jobs and the instruments they had to use thirty years ago, and how the technology has moved on since. A critical step had been interfacing of various pieces of apparatus to cheap PCs, "running in the face of management policy who preferred 'more standard' computer suppliers of the time". His work involved looking at superlattice structures, and the interfacial chemistry of Ga complexes. The accuracies of the structures were then analysed using the dynamical theory and reciprocal space mapping to see whether the materials would be suitable to make

Reports - Spring Meeting 2006 – contd.

superconductor devices. These devices could be used as quantum dots (semiconductor nanocrystals) and then made into fibre optics for faster internet connections. Paul also included some work with a laboratory X-ray source on protein crystal perfection. He finished his talk with the analysis of polycrystalline thin film materials.

The second speaker of the session was **Dr. David Beveridge**, and his talk was on *The precipitation of pigment red 57:1 from homogenous solution for X-ray powder diffraction*. David described the synthesis that was involved in producing the pillar box red pigment. The morphological properties of the pigment looked more crystalline than expected. So, with the help of powder diffraction, it was confirmed that the pigment was indeed crystalline. The red pigment is mainly used for colouring oil-soluble products such as Make-up, powders, lipsticks and foundation creams.

Dr. Mary Vickers gave a lecture on X-*ray diffraction at Materials Science, Cambridge*. Mary talked about the typical samples that are analysed using powder diffraction such as metals, ceramics, and carbon nanotubes. She mentioned the Rietveld refinement and the quantitative phase analyses and cell parameters in steel. Mary has also carried out experiments using Small Angle X-ray Scattering (SAXS) on thin film materials like polyurethane, which is medically important in replacing discs in the human spinal cord. Staying on the topic, Mary highlighted the weakness of SAXS when it comes to identifying water molecules in cellulose fibres. The talk ended with a nice discussion on how students can be encouraged to learn more about X-ray diffraction.

Helal Ahmed
The University of Manchester.

Chris Staddon - *University of Nottingham*
Chris started by giving an overview of the semiconductor material used in the electronic industry Si etc and the described the materials that were being used for development of commercial devices at Nottingham using mainly groups III&V in the periodic table plus N and Mn (for spintronics.) Chris gave a very interesting presentation on ways in which XRD is used to categorise a variety of thin multi-layer semi-conductor materials that are being developed at Nottingham for commercial applications. He described how these samples are grown by Molecular beam expitaxy (MBE) using high vacuum growth chambers in the Physics Dept. Examples were given of how the Crystal quality of the semi-conductors could be measured and how the fractions of hexagonal and cubic phases can be determined. For ferromagnetic semiconductors where the Electron spin (magnetic properties) are important.

Andrew Hodge - *BP*
Andrew explained about the development of XRD at BP and its applications in catalyst development, refining and marketing (engine deposits), and mineral semiquantitative analysis for exploration. A potted background to BP was given as context to several moves of equipment, and Andrew is now the sole person performing XRD in BP.
The best equipment is a new D8-TXS (rotating anode) and Vantec detector for super fast catalyst work. It is also interfaced with an In situ reaction cell using various reactive gases at temperature to mimic conditions inside chemical reactors. An old D5000 provides back up.

Martin Gill, *Imperial College*
Martin gave us a nice slide show of his travels in India and also talked about the perils of not getting analysis before you stick your million quid black & decker into the ground……
Loosing drill strings is very expensive.
Drilling out 3km at angle of 72° - in shales off Mumbai.
Analysis of clay layers. Clay trends different land masses - clays from Basalt. Smectite rich clay - weak drilling close to bedding plane. Not flushing bore hole due to waste pumps breakdowns. Drilling angle too high and too long.
Each drill string cost £1M - cost of analysis £3K

XRD QUIZ.

What Beta filter is used for a Molybdenum X-ray tube?

Know the answer? Then Try our on-line quiz.

Follow the link from the August 2006 web Newsletter page.

Can you think of some good questions for another quiz? If so send them, with the answers, to the web editor.

X-RAY FLUORESCENCE (XRF) PAGE

XRF Meeting 10th May 2006. Our meeting at the British Geological Survey (BGS), Keyworth, Nottingham on the 10th May 2006 was a great success. This joint meeting with the Royal Society of Chemistry (RSC) Atomic Spectroscopy Group was attended by 70 delegates, some appearing on the photograph at the top of this page.
We thank; Ametek –SPECTRO, Analysco, BrukerAXS, HORIBA Jobin Yvon, PANalytical, Rigaku, and Thermo Electron Corporation who all sponsored the meeting. Thanks also to our host BGS for use of the facilities and an excellent buffet lunch and the speakers, chairs and guides shown in the photograph below.

A detailed report on the meeting with more photographs is available on our XRF web pages. Just Google BCA IG and click the XRF link.
We hope to repeat this success with another joint meeting on 14th May 2008!

NEXT MEETING:

17 - 19 April 2007 BCA Spring Meeting.
This meeting at the University of Canterbury has a full three day parallel string of XRF content. Sessions include: a workshop, semi-quantitative, calibration standards, thin films, applications including cultural heritage and environmental issues. There will be an Exhibitors forum allowing suppliers to make you aware of their latest offerings, followed by an evening buffet with the opportunity to talk to vendors at their exhibition stands.
Call for papers: please review the more detailed information on the web and consider offering a talk at the meeting. We will have some short slots for the less experienced to present their first talk. If you feel you have something to offer that won't fill a slot then why not present it in a **poster** at the meeting? There is a prize for best poster and some meeting **bursaries** are available to BCA members.

WEB Newsletter: View our web only XRF Newsletter. The latest edition is (Vol 3 Aug 2006). Please add your name to our distribution list.

XRF planning group: We are pleased to announce that **Ros Schwarz** has agreed to become a member of our XRF planning group and is organising some of the 2007 meeting sessions. The contact details for the XRF group are on the web page – click contacts at the top of the page.

Suppliers List: With your help we plan to build a list of links to XRF suppliers on our web pages. This is very much a work in progress, but please help us by submitting details of suppliers you have found to be useful in your XRF work. We hope that this will build into a very useful resource to help users source equipment, services and consumables. See our Vol 3 web newsletter for details.

Dates for your Diary:
17-19th April 2007 BCA Spring Meeting with a full XRF programme & Exhibition.
14th May 2008 Joint BCA/RSC XRF meeting.

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2006-2007

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