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Newsletter

Autumn 2010

FROM THE CHAIRMAN

Welcome to the Physical Crystallography Group-Structural Condensed Matter Physics Group Autumn 2010 Newsletter. This newsletter, as always, contains an interesting mix of reports on past meetings, news from within our community and invitations to future events. I hope that you will enjoy browsing though it.

As I write this funding cuts are threatening ominously and uncertainty is the order of the day at our centrally funded facilities. This makes it extremely difficult to plan for this year – let alone further ahead – and can easily discourage us. However, it is also a new academic year, with new enthusiastic graduate students arriving and new research projects to initiate. I hope that this year will see more positive outcomes than anticipated and more high quality crystallography from our community. Since I last wrote we have enjoyed another excellent BCA Spring meeting, several of us have been to various crystallographic conferences over the summer. We are also preparing for our Winter Meeting in November, with another Spring Meeting next April and the IUCr Congress in Madrid next summer; somewhere in between these meetings we should also plan to do some work!

Last April saw us gather in Warwick for the BCA Spring Meeting. I felt that the PCG-SCMP sponsored sessions were particularly strongly supported, with high-quality speakers and good audiences throughout. A more detailed meeting report is given later in this newsletter, including a report on the satellite meeting on correcting PDF data, but I would like to thank all of those who helped with the organising of our sessions and in particular to thank Matt Tucker who not only worked hard to make the PDF workshop work so well, but also stood in for me at the Conference Dinner so that I was able to return home to celebrate my 20th Wedding Anniversary. I am told that he did a very good job in introducing our PANalytical Thesis prizes winner to the conference. Indeed one of the highlights of the meeting for me was the session where the PCG's Physical Crystallography and CCG prize-winners delivered their lectures. Christoph Salzmann won the PCG prize this year and he gave a lecture on his high-pressure studies of ice phases. This was followed by Stephen Moggach, this year's CCG prize-winner, who also talked about high-pressure crystallography and his research into a variety of chemical systems including metal organic frameworks. It was a very well attended session and showcased the strength of young talent within our community.

This year's PANalytical Thesis prize went to Helen Maynard-Casely for her work on the planetary mineral methane. We look forward to her speaking about her work at our Winter Meeting and are grateful to PANalytical for their continuing sponsorship of this award. Incidentally Helen is currently working as a researcher for this year's Royal Institution's Christmas Lectures "Size Matters" with Mark Miodownik to be shown on BBC Four; hopefully Helen will have squeezed in some crystallographic content to the lectures. If you, or one of your thesis students, may be eligible for the 2011 PANalytical Thesis prize then please see details of how to apply for the prize later in the newsletter – our previous recipients can attest to how valuable it has been for their future careers.

Our Winter Meeting will follow the same popular format of the previous two years, an overnight meeting at Coseners House, Abingdon in conjunction with the ISIS Crystalography User Group Meeting. The meeting will be on Monday and Tuesday 15th/16th November with an all-encompassing theme "Current Research in Physical Crystallography". We are in the process of completing the programme, but already have an excellent set of speakers who have agreed to come to talk to us; further up to date details are found on our web-pages www.pcg-scmp.org/Meetings/Winter2010 including a link to a page where you can register on-line. The meeting is again supported by ISIS Crystallography Group and the SCMP Group

of the Institute of Physics and I look forward to seeing you in Abingdon in November.

Next year will see a number of changes within the PCG-SCMP Committee; both Ivana and I will have served our allowed tenures as vice-Chair and Chair and Matt Tucker will gradually transfer his responsibilities to Kirsten Christiansen. We therefore are looking for people to take on the chairing roles within the PCG-SCMP group. If anyone has any suggestions as to who might be suitable (including self-recommendations!) then please get in touch with me or our Secretary, Matt Tucker. Elections will take place at the AGM during the BCA Spring Meeting in April 2011.

Best wishes

David Keen
PGG-SCMP Chairman

ANNOUNCEMENTS

PANalytical Thesis Prize 2011

Call for Nominations

The Physical Crystallography Group is pleased to invite entries for the PANalytical Thesis Prize in Physical Crystallography. The prize will be awarded for the best use of techniques or methods of Physical Crystallography in a successfully examined thesis submitted in the period from 1st September 2009 to 31st December 2010.

To be eligible for the prize, candidates must be a member of the Structural Condensed Matter Group of the IoP and/or the British Crystallographic Association (BCA). Non-members may enter the competition but will be required to join the BCA/PCG at the student rate to progress their nomination further (current rate £10 per annum or £27.50 for 3 years of the PhD degree).

To enter the competition, candidates must submit:

(a) a copy of the thesis in electronic format.

(b) a personal statement of not more than 500 words explaining why the thesis should be considered for the prize and including a clear description of the role of Physical Crystallography (as defined on the website www.pcg-scmp.org or otherwise) in the research.

(c) the names and contact details of two academic referees, one of whom may be the thesis supervisor, who will be able to comment on the thesis research of the candidate.

In order for a thesis to be eligible for the award, the Physical Crystallography element must be central to the work of the thesis, which must also demonstrate a context over and above structural work for its own sake.

Nominations for the prize must be submitted to the PCG-SCMP Chair, Prof. David Keen (david.keen@stfc.ac.uk), by 31st January 2011 and the Prize will be awarded at the 2010 BCA Spring Meeting at Keele University, 12-14th April 2011.

Vacancies on the PCG-SCMP Committee

Call for Nominations

Vacancies for the positions of Chair and Vice-Chair are arising on the PCG-SCMP Committee. Nominations for these positions are invited and should be sent to the current Honorary Secretary/Treasurer, Matt Tucker (matt.tucker@stfc.ac.uk).

Nominations should include the name of the proposer, the name of the seconder and the nomination acceptance by the nominee, confirming his/her willingness to take on the leadership responsibilities for the group, and to contribute to the Committee efforts by actively participating in BCA and PCG-SCMP meetings and meeting organisation. Informal enquiries about the group officers' roles should be directed to the current Chairman (david.keen@stfc.ac.uk).

Elections for these positions will be held at the Annual General Meeting of the PCG-SCMP, which will be held during the BCA Spring Meeting at Keele University, 12-14th April 2011.

FUTURE EVENTS

Meeting Calendar

- PCG Autumn Meeting and ISIS Crystallography Users Meeting, 15-16th November 2010, Cosener's House, Abingdon

- Condensed Matter and Materials Physics (CMMP 09), 15-17th December 2009, Warwick University
- BCA Spring Meeting, 12-14th April 2011, Keele University

PCG Autumn Meeting, 15-16th November 2010, Cosener's House, Abingdon

Following the success of the format used the last couple of years, PCG Autumn meeting will take place at the Cosener's House, Abingdon, 15-16th November 2010 (lunchtime-to-lunchtime), in conjunction with the ISIS Crystallography Users Meeting.

Registration for the meeting is free. For participants working at UK-based research institutions who attend both meetings or the Users meeting only, the usual allowable travel costs and overnight accommodation will be reimbursed. A limited number of rooms have been pre-booked at the Cosener's House for the meeting. The rooms will be allocated on a first come first served basis.

The meeting is themed "Current Research in Physical Crystallography". We hope that the meeting will be of interest to a wide audience, from experienced researchers to PhD students. Confirmed scientific presentations include:

Steve Bramwell (UCL)

Eddie Cussen (Strathclyde) "Examining Lithium Ion Mobility in Crystalline Solids"

Mike Hayward (Oxford) "Breaking Inversion Symmetry with Cation Order – New Route to Multiferroic Materials?"

Joe Hriljac (Birmingham)

Helen Maynard-Casely (RI) - PCG PANalytical Thesis Prize winner 2010

Pam Thomas (Warwick)

Richard Walton (Warwick) "Time-Resolved Structural Studies of Flexible MOFs"

Andrew Wills (UCL)

A poster session will take place in the evening of 15th November 2010.

The full scientific programme and the timetable will be posted on the PCG-SCMP wiki (www.pcg-scmp.org).

To register for the meeting, please follow the link given at www.pcg-scmp.org.

BCA Spring Meeting, 12–14th April 2011, Keele University

The BCA Spring Meeting 2011 will take place from Tuesday 12th April to Thursday 14th April 2011 at Keele University. PCG sessions at the meeting will include:

- New Developments at Diamond
- Local Structure
- High Pressure and Energetic Materials
- Time resolved structural science
- Cultural Heritage

The deadline for abstract submission for oral contributions is 29th October 2010, while poster abstracts can be submitted until 4th February 2011.

The scientific programme and further details about the conference will appear at: http://crystallography.org.uk/spring-meeting-2011.

NEWS

Prizes and awards

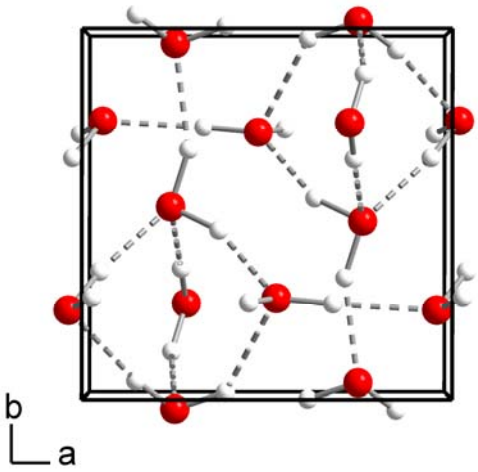
Physical Crystallography Prize 2010

The Institute of Physics sponsored Physical Crystallography Prize 2010 was awarded to Dr Christoph Salzmann, Lecturer in Physical Chemistry at Durham University, for his work on Ice XV and other forms of solid water.

Christoph Salzmann (Durham University, Department of Chemistry), winner of the 2010 Physical Crystallography Prize and Dave Keen, Chair of the PCG-SCMP Committee

The water molecule exhibits immense structural versatility and complexity in its condensed forms in which the H_2O molecules form extended hydrogen bonded networks. The many phases of ice can be grouped into high- and low-temperature pairs. Hydrogen-disordered phases exhibit, subject to the Bernal-Fowler ice rules, orientational disorder of the water molecules, whereas for their hydrogen-ordered counterparts only the energetically most favoured orientations are found. The transition from hydrogen disorder to hydrogen order and the associated decrease in entropy is expected to occur upon cooling. However, the sluggish kinetics of molecular reorientation at low temperatures make the observation of these phase transitions difficult – in most cases, hydrogen disorder is frozen-in upon cooling.

Dr. Salzmann and co-workers showed that doping with hydrochloric acid helps maintain dynamic states to low enough temperatures, so that the phase transitions from hydrogen-disordered ices V, XII and VI to their hydrogen-ordered counterparts ices XIII, XIV and XV can be observed.



Unit cell of ice XV

The discovery of ice XV was of particular significance since it has its own region of stability in the phase diagram. Using powder neutron diffraction, it was discovered that ice XV is antiferroelectric which is in clear disagreement with theoretical calculations predicting a ferroelectric structure as the energetic ground state. The crystal structures of the new ordered phases are therefore important benchmarks for testing the quality of the computer models of water.

The crystallographic studies were complemented by calorimetry and Raman spectroscopy which gave detailed insights into the kinetics of the ordering transitions and the hydrogen bonding properties of these exotic forms of ice.

PANalytical Thesis Prize 2010

The PANalytical Thesis Prize in Physical Crystallography 2010 was awarded to Dr Helen Maynard-Casely (Edinburgh) for her thesis entitled “On the Prediction, Rationalisation and Discovery of New Crystal Forms”.

The main contribution of Helen’s thesis was the solution two high-pressure phases of methane from diffraction data collected in situ, using a combination of x-ray and neutron diffraction diffraction techniques. Both structures were revealed to be more complex than previously thought, departing greatly from the close-packed structures that were expected. It is hoped that now these crystal structures are established, the physical properties of methane at conditions of the Uranian and Neptunian interiors can be constrained more accurately.

Helen Maynard-Casely (Edinburgh) receiving the PANalytical Thesis Prize 2010 from Dr Paul O’Meara of PANalytical

RECENT EVENTS

BCA Spring Meeting, 13–15th April 2010, Warwick University

The BCA Spring Meeting 2011 featured 19 scientific sessions, including four PCG symposia:

- Electron diffraction (organised by Kirsten Christensen)
- Resonant X-ray diffraction (organised by Peter Hatton)
- New approaches to structure solution (organised by Dave Keen)
- PDF: local structure (organised by Matt Tucker)

The PCG Plenary lecture was given by Lynne McCusker (ETH Zurich) and chaired by David Keen. Lynne outlined a 'witch's brew' of techniques available for structure solution from powder diffraction data in her talk entitled 'Polycrystalline Materials, Powder Diffraction, Electron Microscopy and Charge Flipping – A Remarkable Brew. '

Dave Keen with the PCG Plenary speaker, Lynne McCusker (ETH Zurich)

Lynne demonstrated the benefits and drawbacks of electron diffraction compared to X-ray diffraction with reference to complex zeolitic structures. Lynne also highlighted the advantages of repartitioning data midway through a charge flipping refinement where no symmetry is assumed.

Samantha Callear (ISIS)

The first PCG session of the 2010 BCA Spring Meeting, entitled "Resonant X-ray Diffraction" was chaired by Peter Hatton (Durham University) and gave a fascinating demonstration of the kind of complex systems that can be investigated using resonant diffraction and other complementary techniques. Manuel Angst (Forschungszentrum Jülich) began the session with an interesting talk on the "Interplay of electronic and structural degrees of freedom in lutetium ferrite" which focused on the magnetoelectric multiferroic $LuFe_2O_4$. Manuel explained how resonant X-ray diffraction at the FeK edge had been used to confirm charge ordering of Fe^{2+} and Fe^{3+} cations and explain its antiferroelectric structure, whilst polarised neutron diffraction had been used to investigate orbital ordering in the material.

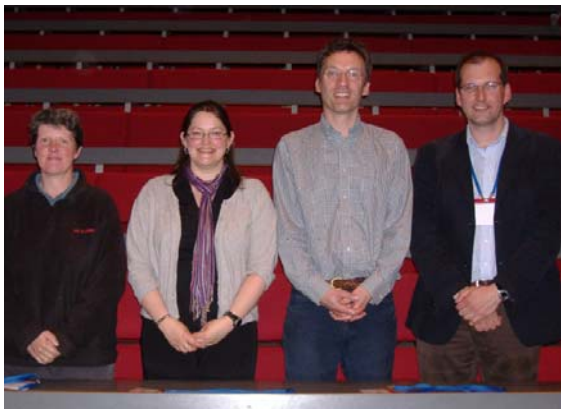
Paolo Radaelli (Oxford University) followed with a fascinating talk entitled "X-ray and neutron studies of multiferroics and frustrated magnets" which showed how subtle structural effects can be elucidated using resonant scattering. Paolo began by looking at the delafossite $AgNiO_2$ and demonstrated how resonant scattering at the Ni K edge had revealed charge disproportionation of nickel to Ni^2+ and Ni^4+ which had helped to explain the magnetic structure of the material. He then went on to discuss some RMn_2O_5 multiferroic materials and how their domain structures had been investigated using neutron spherical polarimetry and resonant scattering.

Speakers at the Resonant X-ray Diffraction session: Manuel Angst, Paolo Radaelli and Sean Langridge, with the session organiser Peter Hatton

The final talk of the session was given by Sean Langridge (ISIS) under the title "Observing interfacial magnetism in nanoscale films". Sean spoke about some FeRh multilayered systems with a magnetostructural phase transition which was investigated using neutron and resonant X-ray scattering and magnetic dichroism techniques. These nanomagnetic systems are of considerable technological importance and can help in the understanding of magnetic behaviour in other systems of reduced dimensionality.

Emma McCabe (Durham University)

The New Approaches to Structure Solution session, chaired by David Keen, was started by Sarah Lister (University of Durham) whose talk entitled 'The Use of Complementary Techniques in Structure Solution from Powders' introduced the benefits of using NMR to probe the local structure. Discussing the $(MoO_2)_2P_2O_7$ system which forms superstructures, the information gained from NMR studies was used to aid structure solution. Following this, Maryjane Tremayne (University of Birmingham) discussed the use of a new direct space global optimisation technique for structure solution known as differential evolution in her talk 'Structure Solution of Multicomponent Systems from Powder Diffraction Data.' Using co-crystals as examples, Maryjane demonstrated how the differential evolution technique offers a fast and robust method for convergence of powder X-ray diffraction refinements (from laboratory data) by 'exterminating' three-quarters of the solution minima population. The session was finished with a talk by Paul Midgley entitled 'Towards Routine Structure Solution using Precession Electron Diffraction.'



Speakers at the New Approaches to Structure Solution session: Maryjane Tremayne, Sarah Lister and Paul Midgle, with session chair Dave Keen

Paul discussed the use of precession in electron diffraction experiments and noted that although the obtained intensities are not strictly kinematical, they can be used for structure solution. The effectiveness of this technique was shown to be improved when employed in combination with a new charge flipping algorithm that includes the crystal symmetry.

Samantha Callear (ISIS)

Andrew Goodwin gave the first talk of the Pair Distribution Function (PDF): Local Structure session, entitled 'From shells to solar panels – 'solving' the structures of amorphous materials using PDF'. He presented a range of materials and showed that by modelling the PDF for each of these, information on local structure could be obtained. In one example, a zeolitic imidazolate framework material (ZIF), underwent a thermally-induced transition to an amorphous phase and then surprisingly re-ordered at higher temperatures to give a new denser crystalline phase. PDF analysis showed that the sample actually retained the same local environment although the long range order differed between the crystalline polymorphs. Andrew also demonstrated the possibility of structure 'solution' using amorphous $CaCO_3{\cdot}H_2O$ as an example. Starting with an ordered atomic configuration and introducing disorder produced the same PDF that was obtained when starting from a random configuration of atoms and introducing order instead. The resultant atomistic model could be regarded as a 'solution' of the amorphous structure.

Speakers at the Pair Distribution Function (PDF): Local Structure session: Daniel Shoemaker, Samantha Chong and Andrew Goodwin, with session chair Matt Tucker

Daniel Shoemaker's talk 'Locating atoms in disordered crystals' highlighted exactly why 'interesting things happen when crystals are disordered'. The complex $Bi_2Ti_2O_7$ possesses a cation 'off-centring problem' where bismuth and oxygen atoms are displaced from their ideal positions. This disruption on long range ordering meant accurate information on the structure could not be obtained from Rietveld analysis alone. Reverse Monte-Carlo (RMC) modelling of the PDF produced a 'snapshot' of the local structure showing the instantaneous deviations in bismuth and oxygen positions. Daniel highlighted another advantage to the PDF method – in the complex $CuMn_2O_4$,

disordered copper and manganese atoms adopt both octahedral and tetrahedral geometries. Rietveld refinement would constrain the coordination geometries of manganese and copper to be the same but this is not the case when using the PDF approach. In fact it predicted the existence of Cu3+, which occurs via charge disproportionation in order to avoid Jahn-Teller distortions. Samantha Chong gave the final presentation of the session 'Investigating disorder in a pure Bi A site perovskite using total scattering and RMC modelling'. Samantha presented a perovskite structure that featured a mixture of titanium, iron and magnesium occupying the 'B' site. However, similarly to the problem described by Daniel, Rietveld refinement constrained all B site atoms to be the same, ich led to incorrect bonding coordination according to bond valence sums analysis. Using the RMC method but utilising bond valence sums as constraints on the atomic configuration, the disorder in the system could be more accurately modelled. The results showed there was no significant long or short range ordering for the B-site atoms and that the B-site itself had little structural consequence on the Bi-O bonds.

Nicholas Funnell (University of Edinburgh)

PDF workshop: Local structure, data corrections matters

Following the success of the PDF and Reverse Monte Carlo analysis workshop held before the 2007 BCA spring meeting, this year the PCG organized a hands on workshop: Local structure, data corrections matters. The aim of this workshop was to teach the participants how to collect and correct data for Pair Distribution Function and total scattering analysis.

The workshop started at 2:30 pm on the 15th of April (directly after the main BCA Spring meeting in Warwick) and finished 12:30 pm on the 16th April. During the first afternoon Prof Alan Soper introduced the key concepts and theory of neutron total scattering measurements. He then lead a hands-on session where people could try the GUDRUN program he has written to produce corrected neutron total scattering data. Prof David Keen then gave a talk titled 'Total Scattering Data Correction Tricks of the Trade' where he discussed the practical problems that people may encounter and their solutions. The first day finished with more discussion over a very nice dinner and some wine.

The second day began with Prof. Alan Soper introducing the key concepts and theory of X-ray total scattering measurements. Then after coffee Dr Emma Barney led a hands-on session using the GUDRUN-X program to produce corrected X-ray total scattering data.

Participants of the PDF: Local structure, data corrections matters workshop

The workshop was very successful and useful, with the ~30 participants enjoying themselves and hopefully learning to use the total scattering technique in their research. If you would be interested in attend any future workshops on this topic please email Matt Tucker (matt.tucker@stfc.ac.uk).

Matt Tucker (ISIS)

ACKNOWLEDGEMENT

Many thanks to everyone who contributed to this issue of the PCG-SCMP Newsletter.

Ivana Evans, Durham

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