
COMMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

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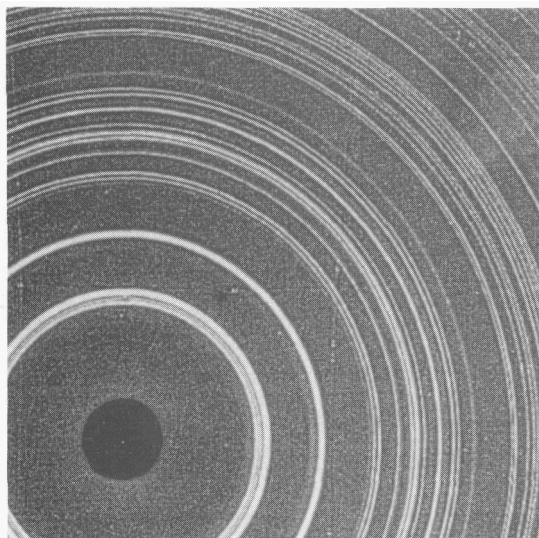
HIGH-PRESSURE POWDER DIFFRACTION AT SRS DARESBUURY USING AN IMAGE-PLATE DETECTOR

The application of high pressure brings about a great richness of variation in the structures and physical properties of materials of all kinds. To advance our understanding of high-pressure phenomena, it is necessary to determine the relevant crystal structures accurately under pressure. This has been achieved in some cases by single-crystal methods, but it is not generally possible to work with single-crystal samples above 10-20 GPa (100-200 kbar) because of the (small) sample size and the need to have sufficient angular access to measure a large number of reflections. Also, many systems undergo a pulverising first-order transition at a pressure below 10-20 GPa, and powder diffraction is then the only way to study the high-pressure phases. But success with powder methods has been limited so far.

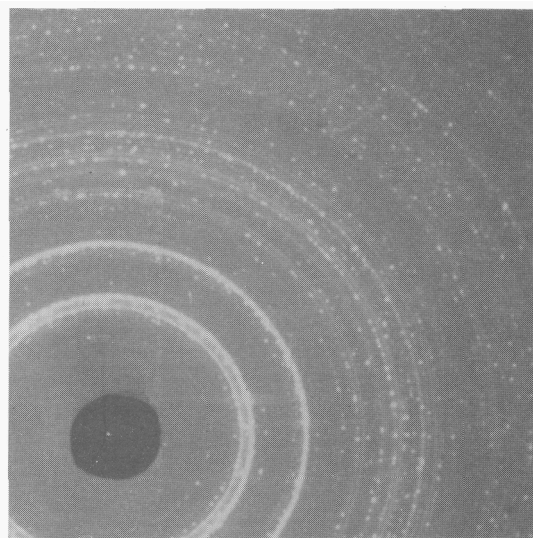
The best device for holding a powder sample at pressures for diffraction measurements is a diamond-anvil cell (DAC). The sample is placed in a hole in a piece of metal (the gasket) which is squeezed between two opposed flat-tipped diamonds. Usually a liquid or soft medium is mixed with the sample to give reasonably hydrostatic conditions. Even at a relatively modest

pressure of 10 GPa, the sample may be only 150 μm across and 50 μm thick, illuminated by a beam <100 μm across to avoid scattering from the gasket. Hence sample volumes are at least a factor of 1000 smaller than for ambient-pressure studies. Not only does this give weak signals, but also poor powder averaging due to the small number of diffracting crystallites.

Almost all powder-diffraction work to date has been done by energy-dispersive techniques. Use of the full 'white' beam from a synchrotron compensates for the small sample volume, and data collection rates can be quite fast. But tight collimation is required and this makes peak intensities particularly vulnerable to the problem of powder-averaging. Also, energy-dispersive patterns contain unavoidable contaminant peaks and the resolution is limited to about 1.0 % by the properties of the solid-state detector. For these reasons, energy-dispersive studies have focused on unit-cell dimension measurements and the identification of structure types, rather than full refinement of crystal structures (though there are now some promising developments towards making this possible).



(a)



(b)

Fig. 1. Powder patterns from InSb in a diamond-anvil cell, (a) in its first high-pressure phase at 3.5 GPa, and (b) partially recrystallised to a second phase at the same pressure.

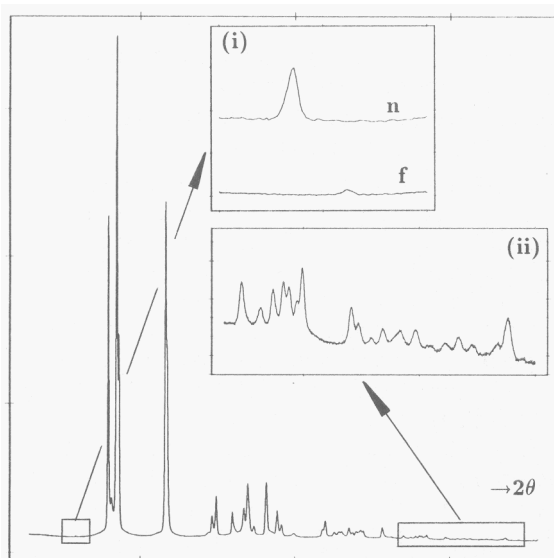
A few years ago, workers at the Photon Factory (PF) made a breakthrough [1] by exploiting the recently developed image-plate detector. This is a sheet of phosphor, normally about 20x25 cm² in area, in which incident X-ray photons generate trapped electron states. Subsequent scanning with a He-Ne laser causes stimulated emission of (blue) light, which is collected and converted to a digital image of the incident X-ray diffraction pattern on a pixel size of typically 100x 100 μm². The intensities in the final image vary linearly with the number of X-ray photons incident on the image plate over a dynamic range of some 10⁵. As well as having this high dynamic range, and high sensitivity, the phosphor has a very low intrinsic background, making it an ideal detector for weak signals. The idea developed at the PF was to record the central part of an angle-dispersive powder pattern (about ±30°(2θ) x 7" with a pressure cell) and then integrate around the segments of the powder rings in the digitised image, much enhancing both the signal and powder averaging. This overcomes the problems of the small sample volume and makes it possible to exploit the advantages of angle-dispersive methods for high-pressure structural work.

Over the past year, we have considerably developed this technique at SRS Daresbury, with the eventual objective of carrying out full Rietveld refinement of high-pressure structures. To this end, we have concentrated on getting completely contaminant-free patterns, and we have departed from conventional high-pressure powder-diffraction techniques by using only DAC's with full conical apertures (giving complete powder rings to 2θ = 40") [2]. We have also developed general software for optimised integration of the 2 d image [3].

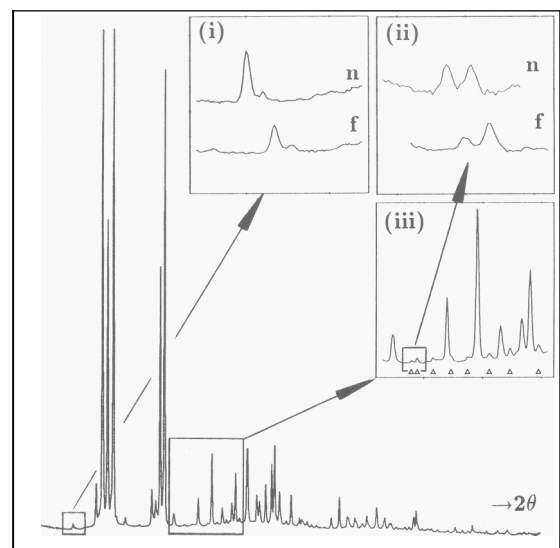
Fig. 1a has been digitally enhanced to bring out weak features. It shows part (approximately a third) of a 2-d image recorded on our system from a high-pressure phase of InSb, at about 3.5 GPa (35 kbar), using a wavelength of ≈0.48 Å from a channel-cut Si(111) monochromator on the wiggler station 9.1 at SRS

Daresbury. Just discernible at 0.7 of the radius of the first strong line is a very weak low-angle line. The integration of the whole of the original (unenhanced) image is displayed in Fig. 2a, and the very weak low-angle line is enlarged in the lower profile of inset (i). The intensity of this line is only 1/1000th of the adjacent strong lines and arises from the (small) difference in scattering between In and Sb. This can be confirmed by repeating the measurement at 0.455 Å, much nearer the In K-edge. As shown in inset (i), the intensity of the line increases significantly when the wavelength is changed from being far (f) from the edge to near (n) it. (The change in wavelength shows up in the small shift of the peak to lower 2θ) We believe this to be the first reported observation of anomalous scattering effects in high-pressure powder diffraction. Detecting such a weak feature depends not only on the characteristics of the image-plate detector, but also on minimising all sources of background and integrating over complete powder rings. At the position of this particular line, the integration is over 1000 pixels and this is crucial in reducing the background noise to the required level.

Inset (ii) of Fig. 2a shows (enlarged) the well-resolved diffraction pattern extending to high angles - beyond the range of the part of the image shown in Fig. 1a. At a normal working sample-to-plate distance, D, of 100-300mm, the width of the lines is determined by the resolution of the read-out process from the phosphor, and so the angular width reduces with increasing D. Typically, the FWHM is 0.08" at D = 250 mm, falling almost linearly to 0.04" at 500 mm, which compares favourably with all but the highest resolution techniques. Generally there is some pressure broadening in compressed samples, and it is doubtful if there are many cases where it would be useful to have higher resolution than the image plate can give. As can be seen, the integrated profile has symmetric peak shapes. This, the extent and resolution of the pattern, and the uniform background allow good results to be obtained in Rietveld refinement.



(a)



(b)

Fig. 2. (a) Integration of the InSb pattern in Fig. 1a and (b) integration of the pattern obtained after complete recrystallisation to the second phase in Fig. 1b.

Fig. 1b shows the emergence of a second high-pressure phase in InSb, without increasing the pressure from that of Fig. 1a. The smooth lines of Fig. 1b are the same as those in Fig. 1a; the spots are from a new phase, recrystallising under pressure. As can be seen, some spots lie on the lines of the first phase and others are in new positions. We have successfully indexed all the 'spotty' lines on a unit cell closely related to that of the first phase, but much larger. In time, the proportion of the 'spotty' phase increases, and its lines become much more continuous. An integration of a pure 'spotty'-phase pattern is shown in Fig. 2b - again going beyond the range of the part of the image shown in Fig. 1. It turns out that the strongest lines of this pattern correspond to the previously published high-pressure phase of InSb, but we have been able to detect a large number of very weak reflections, such as those marked (A) in inset (iii), which show the true unit cell to be 12 times the volume of the one proposed in the literature. We can show that this phase, too, is ordered: insets (i) and (ii) in Fig. 2b illustrate some of the changes in relative intensity seen as the wavelength is brought near (n) to the In K-edge. In this case, having the full 2-d image was crucial in separating and interpreting the two phases, as well as in giving low noise in the integrated profile.

The combination of the image plate, with minimal background and careful collimation (to remove contaminant scattering), conical-aperture cells (to give full 2-d images) and good data analysis clearly offers a great deal for the future of high-pressure crystallography

on synchrotron sources. It is now possible to contemplate accurate structural work at very high pressures and/or on very weakly scattering samples on present and next-generation synchrotrons. Also the success in exploiting anomalous scattering opens up the possibility of studying other electronic effects and electron density variations under pressure.

The project at Daresbury is the work of a group from The University of Edinburgh (Richard Nelmes, Peter Hatton, Malcolm McMahon, Ross Piltz and Jason Crain) strongly supported by Daresbury Laboratory and its staff (particularly Bob Cernik, Graham Bushnell-Wye, Mike Miller, Chris Hall and Alf Neild) and with a research grant from the UK Science and Engineering Research Council. The initial development of the project owes much to Prof. H. Iwasaki, Prof. Y. Fujii, Dr. O. Shimomura, Dr T. Kikegawa and their colleagues through access to their image-plate system at the Photon Factory and assistance in using it.

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- [2] R.J. Nelmes, P.D. Hatton, M.I. McMahon, R.O. Piltz, J. Crain, R.J. Cernik and G. Bushnell-Wye, *Rev. Sci. Instrum.* **63**, 1039 (1992).
- [3] R.O. Piltz, M.I. McMahon, I. Crain, P.D. Hatton, R.J. Nelmes, R.J. Cernik and G. Bushnell-Wye, *Rev. Sci. Instrum.* **63**, 700 (1992).

R.J. Nelmes

IN SITU NEUTRON POWDER DIFFRACTION AT THE ILL - GRENOBLE

The 57 MW High Flux Reactor of the Institut Laue Langevin at Grenoble has been in operation since 1972 providing neutron beams to about 31 scheduled instruments. When the reactor was shutdown in April 1991 for a major refurbishment, the Institut was operating four powder diffraction instruments. The instruments of the first generation were located on a neutron guide :

D1A was the first successful high resolution neutron powder diffractometer and has been largely responsible for the growth of neutron powder diffraction over the last two decades [1]. However, the long data collection times (\approx 12 hours/pattern) required by this instrument preclude its use to investigate time-dependent chemical or physical phenomena.

D1B is a high-flux, medium resolution powder instrument that is equipped with a 400 cell curved position sensitive detector spanning an angular range of 80 degrees. Over the last decade or so, it has been used to develop time and temperature resolved powder diffraction with data collection times of the order of a few minutes [2]; its poor resolution however severely limits the structural information it can provide.

The high penetrability of neutron beams through most elements makes neutron diffraction particularly suited to investigate materials in complex environmental cells and rapid data collection then enables *in situ* investigation of their transformations under various stimulations. This approach has proved to be

particularly valuable in studies of chemical reactions to identify intermediate species or to elucidate the structural mechanism of phase transformations. The development at the ILL of two new powder instruments that were built closer to the reactor to benefit from a higher neutron flux now makes *in situ* experiments much easier: D2B with its 64 detector bank and high resolution ($\Delta d/d \leq 10^{-3}$) permits large structures to be examined in a few hours; D20, although still equipped with a 12.6 degree detector, benefits from a higher flux and/or better resolution than D1B.

The possibilities offered by these new instruments have stimulated much interest in the investigation of chemical reactions and of temperature-induced transformations. A first example is provided by a recent study of the high temperature tetragonal-to-monoclinic transformation of zirconia. Although this transformation has been extensively studied in the past and its basic features elucidated, the details of the structural modifications are not yet understood. Using a mirror furnace, Frey et al. [3] have collected high resolution data on D2B at temperatures up to 1900K to refine the structure of ZrO_2 in the coexistence range of the monoclinic and tetragonal phases. Their Rietveld refinements support a two-step transformation process: first a displacive precursor regime then a martensitic transition. By analysing the line shapes they could further show that microstrains are present in the two phases with a minimum in the coexistence region where large macrostrains develop.

Another example concerns the behavior of alkaline MnO_2 batteries. Manganese dioxide in the crystalline form known as $\gamma\text{-MnO}_2$ has been used in primary batteries (Leclanché batteries) for almost a century. Yet the detail of the structural transformations that take place during battery discharge was not known; in particular the origin of the non-reversibility of the reaction $\text{MnO}_2 + e^- + \text{H}^+ \rightarrow \text{MnOOH}$ that implies that the battery is not fully rechargeable had never been clearly identified. This information could be obtained by carefully monitoring the diffraction pattern of selected $\gamma\text{-MnO}_2$ samples throughout the discharge in an electrochemical cell designed for neutron diffraction; owing to the poor crystallinity of this material the experiment does not require high instrumental resolution but high flux. It was performed [4] on D1B/D20 with two $\gamma\text{-MnO}_2$ samples having different concentration of structural defects (rutile slabs interspersed in a ramsdellite lattice). The results (see Fig. 3) show that

reduction of Mn^{4+} in the lattice planes containing these defects leads to the destruction of the structure of the starting material with the formation of a new phase (pyrochroite) that will not regenerate the initial structure upon recharge. The rechargeability of batteries is thus fundamentally limited by the presence of structural defects.

The HFR is planned to resume operation by 1994. Additional information on the ILL can be obtained from the following address: SCAPRO, Institut Laue Langevin, 156X - 38042 Grenoble Cédex (France).

- [1] A.W. Hewat, *Chemica Scripta* 26A, 119 (1985).
- [2] J. Pannetier, p. 313 in *Chemical Crystallography with pulsed neutrons and synchrotron X-rays*, edited by M.A. Carrondo & G.A. Jeffrey, Reidel (1988).
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- [4] M. Ripert, J. Pannetier, Y. Chabre and C. Poinignon, *MRS Proceedings* 210, 359 (1991).

J. Pannetier

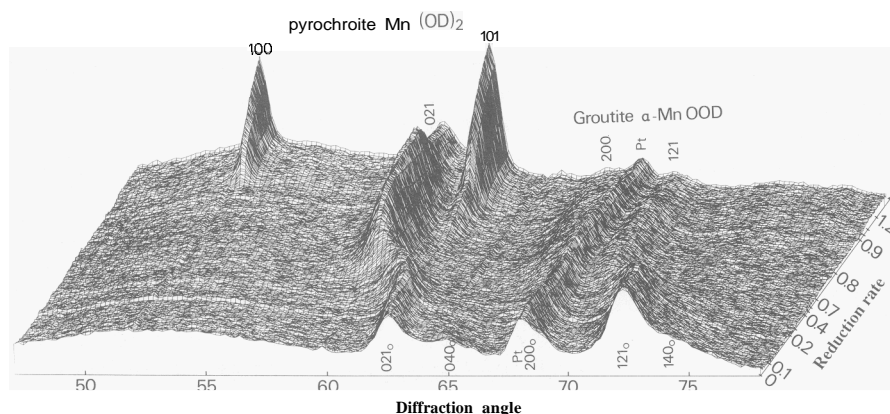


Fig. 3. Evolution of the neutron powder diffraction pattern of a commercial $\gamma\text{-MnO}_2$ sample during discharge in alkaline electrolyte. The high background below the peaks arises from the silica vessel and KOD/7N solution used as electrolyte. The new lines from pyrochroite $\text{Mn}(\text{OH})_2$ that show up in the final stage of reduction indicate a partial breakdown of the $\gamma\text{-MnO}_2$ lattice; they are observed only for samples having large concentrations of defects.

POWDER DIFFRACTION AT ESRF

At the European Synchrotron Radiation Facility a high resolution powder diffractometer will be constructed on a bending-magnet beam line (No. 15). This is a second-wave instrument and is destined to commence operation in 1995. The diffractometer will operate with flat plate or capillary specimens, and various sample environments (cryostats and furnaces) will be provided. The approximate wavelength range will be 0.3-2.5 Å (40-5keV) which includes the K or L_{III} edges of all the elements from Ti to U.

The instrument will be designed to obtain very high 2θ and energy resolution. The target specifications are an instrumental contribution to the peak width of around 0.002° and an energy bandpass through the monochromator of less than 1 eV at 10 keV. Furthermore, with an appropriate detector system very rapid data collection will be possible, in a lower-resolution mode, opening up possibilities for dynamic diffraction measurements.

The high-resolution characteristics of the diffractometer will enhance powder-diffraction studies in a number of areas.

1) With a very small instrumental contribution, the overall peak shape will be dominated by the sample and the effects of crystallite morphology, homogeneity, crystallite size, stresses and strains. The peak shapes will therefore contain a great deal of information about the microstructure of the sample, but may therefore be more difficult to model for a Rietveld refinement of the crystal structure. Nevertheless, with suitable peak-shape functions, the simultaneous characterisation of both microstructure and the crystal structure of powdered specimens will be of importance for many modern materials, such as ceramics.

2) The improved angular resolution of the diffraction patterns will increase the success of the *ab initio* determination of structures from powder data. From the low-angle data the capabilities of indexing much larger unit cells will be improved, and the reduced peak overlap

at higher angles will lead to the solution of more complex structures. With current diffractometers, the severe peak overlap at high values of 2θ reduces the number of reflections whose intensities can be determined well enough for the successful application of direct methods. When combined with new powerful methods of analysis such as maximum entropy, we might expect to solve structures of a complexity currently accessible only by single-crystal techniques.

3) High energy resolution, coupled with the tunability of the incident radiation, will provide a fine tool for probing near X-ray absorption edges to exploit resonant scattering phenomena. As well as differentiating between adjacent elements in the periodic table, one of the most exciting prospects involves correlating the shift in the position of the edge, from the accompanying changes in Δf and $\Delta f''$, with the oxidation state of the element. Hence the diffraction experiment becomes site specific, and information regarding the oxidation state of

an element in a particular crystallographic site may be inferred. The feasibility of such studies has already been demonstrated using current synchrotron sources.

The combined use of neutrons and synchrotron radiation in powder diffraction studies of difficult systems has in the last few years begun to show great promise. The ESRF shares a site in Grenoble with the Institut Laue-Langevin where several powder neutron diffractometers operate. It is to be expected that, with their close proximity, further developments will be made in the application of complementary high resolution neutron and synchrotron-radiation diffraction to complex structural problems in powder crystallography.

Further information can be found in the report on the ESRF Powder Diffraction Workshop, (March 27-28, 1991), edited by Å Kvik.

A.N. Fitch

ENCOMIUMS

Professor R.A. Young has been elected into the Polish Academy of Sciences as a Foreign Member. The election took place at a meeting of the Academy in Warsaw on May 17, 1991. Nine American Scholars were elected at this triennial election. The ceremony of inducting Professor Young into the Academy by Polish Ambassador Kazimierz Dziewanowski took place at the Embassy of Poland in Washington, DC, on Sept. 23.

NEWS FROM ICDD

The results of the Ballot for Election dated 3 Jan. 1992 are presented herewith : **Chairman** Gerald G. Johnson, Jr. ; **Vice Chairman** Jan W. Visser ; **Members-at-Large**, Board of Directors, Walter Eysel, Melvin H. Mueller.

The International Centre for Diffraction Data is pleased to announce the awarding of two Crystallography Scholarships for 1992 : J.M.S. Skakle, Univ. of Aberdeen (UK) and T.P. Hackwell, Univ. College London (UK), have been selected as the recipients by the ICDD Scholarship Award Selection Committee. Janet Skakle is pursuing a PhD degree studying structures of a variety of barium rare-earth titanates using neutron and X-ray diffraction. Timothy Hackwell is conducting graduate research in phase transitions on feldspars.

STATUS OF CPD PROJECTS

Accuracy in Powder Diffraction II

Organisation of the APD II conference at NIST, Md, USA on May 26-29, 1992 is progressing well. The meeting is being jointly sponsored by the JCPDS-ICDD, IUCr, and IUCr Commission on Powder Diffraction. The programme will consist of both invited and contributed papers in sessions devoted to :

- * Phase identification and quantification
- * Accuracy and standards

- * New developments in software and data analysis
- * Profile fitting and microstructural effects
- * Novel applications and structural science
- * New developments in hardware
- * Studies under non-ambient and time-resolved conditions.

In addition, there will be a tribute to the work of Dr W. Parrish, two ICDD Workshops (one on Data Accuracy and another on Indexing) and an open meeting of the Commission on Powder Diffraction. Social events include a Wine and Cheese Mixer, a Conference Banquet and a Reception at the Smithsonian Institution of Washington.

To give you a feel for the depth and scope of the meeting, the list of distinguished invited speakers includes :

J.P. Attfield (Cambridge, UK), J.F. Bézar (Paris, France), D.L. Bish (Los Alamos, USA), H. Boysen (Munich, Germany), J.P. Cline (NIST, USA), B.L. Davis (South Dakota, USA), L.W. Finger (Washington, USA), Y. Fujii (Tsukuba, Japan), H. Hermann (Dresden, Germany), T.M. Holden (Chalk River, Canada), T.C. Huang (IBM Research, USA), J.I. Langford (Birmingham, UK), A. Le Bail (Le Mans, France), D. Louër (Rennes, France), L.B. McCusker (Zurich, Switzerland), R.J. Nelmes (Edinburgh, UK), J.M. Newsam (Biosym Tech., USA), R.L. Snyder (Alfred, USA), J.K. Stalick (NIST, USA), B.H. Toby (Allentown, USA), V. Valvoda (Prague, Czechoslovakia), P.-E. Werner (Stockholm, Sweden), T. Yamanaka (Osaka, Japan), L. Zevin (Beer-Sheva, Israel).

Anyone who wishes to attend and has not yet registered, please send a request to the Local Chairmen, Dr E. Prince or Dr J.K. Stalick, Reactor Radiation Division, Room E151, Building 235, NIST, Gaithersburg, MD 20899, USA. Fax: +1-301-921-9847 E-mail : prince@enh.nist.gov or prince@nbsenh. Depending on the lateness of the request, it may still be possible to submit a late abstract for the meeting.

Intending participants should note that the oral sessions on the last day of the conference (Friday 29) have been arranged to provide an introduction to the Workshop on X-ray and Neutron Diffraction at High Pressure, organised by the High Pressure Group of the IUCr

Commission on Apparatus. This Workshop will be held at the Geophysical Laboratory, Carnegie Institution of Washington and will run from May 29 to 31. For further information, contact Dr L.W. Finger, Geophysical Laboratory, Carnegie Institution of Washington, 5251 Broad Branch Road, NW, Washington, DC 20015, USA. Tel: +1-202-686-2464, Fax: +1-202-686-2419, e-mail : finge@gl.ciw.edu

R.J. Hill
Scientific Programme Chairman

Rietveld Summer School, Cieszyn (Poland), August 13-15, 1992

The school is organized by the Commission on Powder Diffraction, IUCr, Silesian University (Katowice, Poland), Institute of Ferrous Metallurgy (Gliwice, Poland) with the financial support of the IUCr and will be held just after closing the XV Conference on Applied Crystallography. The school will consist of lectures and practical training. Contact : Dr Danuta Stroz, School Secretary, Uniwersytet Slaski, Instytut Fizyki i Chemii Metali, ul. Bankowa 12, 40-007 Katowice, Poland. Phone 59-69-29.

International School of Crystallography : Computational Methods in X-ray Powder Diffraction Analysis

The CPD is co-organizer of this fourth International School of Crystallography to be held in Aswan, Egypt, in January 1993. The CPD Chairman, Prof. R.A. Young, and members J.I. Langford and D. Louër are members of the programme committee. The organizing and local chairman is Prof. Karimat El-Sayed, Physics Department, Faculty of Science, Ain Shams University, POB 8014, Masaken Nassr, Nassr City, Cairo, Egypt.

Powder Diffraction Satellite Meeting, Hangzhou, 1993

A Powder Diffraction Satellite Meeting will be held at Hangzhou, an attractive city some 450 km to the west of Shanghai, immediately after the XVIth IUCr Congress and General Assembly. The probable dates are 31 August to 3 September 1993. I have already received some ideas for the programme, but would welcome further suggestions for topics and speakers and also views on whether or not the meeting should be 'thematic', concentrating on a particular aspect of powder diffraction. If you have not already done so, please send your recommendations to me at my address given in the last page.

J.I. Langford
Programme Chairman

MEETING REPORTS

Proceedings of the First European Diffraction Conference EPDIC 1

EPDIC 1 was held 14-16 March 1991 in Munich (Germany). The proceedings - edited by R. Delhez and E.J. Mittemeijer - contain 150 contributions comprising about 960 pages including numerous figures. They have

just appeared as volumes 79-82 of the series Materials Science Forum (ISBN 0-87849-634-3 ; SFr 420 (ca. US\$ 278) which is published by Trans Tech Publications (Segantinstrasse 216, CH-8049 Zurich, Switzerland or Old Post Road, Brookfield VT 05036, USA).

The present book places emphasis on new (methodological and instrumental) developments and concentrates on (i) the application of (high intensity) synchrotron radiation (for example for structural analysis of thin films), (ii) unravelling of overlapping clusters of peaks, (iii) selection of profile-shape functions for Rietveld analysis and line broadening analysis, (iv) elimination of errors due to measurement limitations (as truncation of the range of measurements) and (v) analysis of structural change in non-ambient conditions.

News from Japan

A group meeting powder diffraction has been held in Hakone, Japan on January 20 and 21, 1992. This group, consisting of 11 members and organized by Prof. Sakata of Nagoya University, exchanged information about the recent uses of the powder diffraction method. The Japanese government provided a scientific fund for the financial years of 1991 and 1992. Topics discussed include materials studies with the Rietveld and the profile fitting methods, a new powder diffractometer for neutron sources, studies with specimens under special conditions, uses of image-plate detectors, TDS correction, *ab initio* structure determination, the maximum entropy method and its computational problems. Some 25 attendants enjoyed a beautiful meeting site of the foot of Mt. Fuji and hot springs. The next meeting will be held by the end of 1992.

H. Toraya

UIMP Summer Course on Materials Characterization by Diffraction Methods, Barcelona, July 15-19, 1991

The course was held 15-19 July 1991 in Barcelona (Catalunya, Spain) thanks to the sponsorship of the 'Universidad Internacional Menendez-Pelayo' and the CSIC. The secretary was Dr J.F. Piniella, with Prof. C. Miravittles and Dr. J. Rius as the academic organizers. There were about 45 students, most of them from Spain but also some coming from the rest of Europe.

The purpose of the course was to provide a general overview of the most significant theoretical and experimental advances in the characterization of crystalline materials by X-ray and neutron diffraction techniques. The course was divided into three parts, *i.e.* single-crystal diffraction, powder diffraction and information sources in Crystallography. To favour contact between lecturers and students, the general plan of the course was two lectures in the morning, two in the afternoon, and a round-table discussion in the evening. The course excursion was to the Montserrat monastery and the visit to two Collbató caves.

The inaugural lecture was given by Prof. Nardelli on general considerations about diffraction crystallography to solve structural chemistry problems. The topics on single-crystal diffraction and information sources were covered by Prof. Beurskens (Nijmegen), Dr Fita (Barcelona), Prof. Germain (Louvain), Prof. Lecomte

(Nancy), Prof. Martinez-Rip011 (Madrid), Dr Rius (Barcelona) and Dr Zuñiga (Bilbao).

The powder diffraction topics were presented by Dr Baerlocher (Zürich), Dr Gali (Barcelona), Dr Louër (Rennes), Dr McCusker (Zurich) and Dr Rodriguez (ILL). Of particular interest to powder diffractionists were the impressive results obtained from the optimal use of laboratory X-ray equipment shown by Dr Louër, as well as the *ab initio* solution, and subsequent restrained Rietveld refinements of zeolite structures from synchrotron data presented by Dr McCusker. **Also** of interest was her explanation of the Squared Patterson method developed by Dr M. Estermann (Zürich) for the treatment of overlapping reflections, with potential for the application of Direct Methods to powder data. The interest in these topics was reflected in the large number of questions arising in the round-table discussions.

J. Rius

X-ray Diffractometry and Spectrometry in Production Quality Control, Brno, Czechoslovakia, November 6-7, 1991

This seminar, organized jointly by COMMET Bmo, SIEMENS Karlsruhe, Czechoslovak Crystallographic Society and Spectroscopic Society under the chairmanship of Dr Jirí Zeman (Bmo), brought together 107 people from different laboratories in Czechoslovakia. The main part of its scientific programme had the form of a series of invited lectures on database systems, X-ray diffraction stress measurement, texture analysis and new software for X-ray spectrometry. The seminar was designed to draw attention to the various possibilities of X-ray diffraction and spectroscopy in the development of new technologies and in environmental protection programmes.

J. Fiala

FUTURE MEETINGS OF INTEREST TO POWDER DIFFRACTIONISTS

Powder diffractionists are invited to send notices of forthcoming meetings to the editor of the next CPD Newsletter, Dr D.E. Cox, at his address shown on the back page.

26-29 May 1992 **Accuracy in Powder Diffraction 11**, National Institute of Standards and Technology, Gaithersburg, Maryland, USA (Dr E. Prince, Reactor Radiation Division, NIST, Gaithersburg, MD 20899, USA).

29-31 May 1992 **Diffraction at High Pressure**, Washington, DC (Dr L.W. Finger, Geophysical Lab., 2801 Upton St., NW, Washington, DC, 20008-3898, USA ; e-mail : finger@gl.ciw.edu).

1-7 June 1992 **IUCr School on Crystallographic Computing**, Veszprem, Hungary. (Dr Kalman Simon, Hungarian Chemical Society, H-1027 Budapest, Fő u. 68, Hungary).

30 July-1 Aug. 92 **Second European Powder Diffraction Conference**, Enschede, Holland (Dr T. Ryan, Philips Analytical, Lelyweg 1, NL-7602 EA Almelo, The Netherlands).

2-7 Aug. 1992 **14th European Crystallographic Meeting (ECM-14)**, Twente, The Netherlands. (Dr H.J. Bruins Slot, CAOS:CAMM Center, Univ. of Nijmegen, The Netherlands).

9-14 Aug. 1992 **American Crystallographic Assoc. Annual meeting**, Pittsburg, PA, USA (M.J. Vair, ACA, P.O. Box 96, Ellicott Station, Buffalo, N.Y. 14205-0096, USA).

9-12 Aug 1992 **XVth Conference on Applied Crystallography**, Cieszyn, Poland (Dr D. Stroz, Inst. Phys. & Chem. Metals, Silesian Univ., ul. Bankowa 12, 40-007 Katowice, Poland).

13-15 Aug. 1992 **Rietveld Summer School**, Cieszyn, Poland (Dr Danuta Stroz, Inst. Phys. & Chem. Metals, Silesian University, ul. Bankowa 12, 40-007 Katowice, Poland).

1-4 Sept. 1992 **Int. Seminar on Structural Investigations at Pulsed Neutron Sources**, Dubna, Russia (Dr. Yu.V. Taran, JINR, 141980

Dubna, Moscow region, Russia, e-mail : bala@lrp04.jimr.dubna.su).

Oct. 1992 **Swiss Society of Crystallography, Annual Meeting**, Basel (Pr H. Stockli Evans, Inst. de Chimie, Univ. de Neuchâtel, 51 av. de Bellevaux, 2000 Neuchâtel, Switzerland).

14-16 Nov. 1992 **1st Crystallography Conference of the Asian Crystallographic Assoc.**, Singapore. Contact : Prof. N. Kasai, Applied Cryst., Osaka U., 2-1 Yamadagaoka Saira, Osaka 565 Japan. For programme info: Dr E.N. Maslen, Crystallography Centre, U. of Western Australia, Nedlands, Perth 6009, WA, Australia.

Jan. 1993 **Fourth International School of Crystallography : Computational Methods in X-Ray Powder Diffraction Analysis**, Aswan, Egypt (Prof. Karimat El Sayed, P.O. Box 8014, Masaken Nassr City, Cairo 11371 Egypt, tel. : 02-2601742 ; telex : Hecor 4054, e-mail : karima@egfrcuvx.bitnet).

29 Mar.-2 Apr. 1993 **BCA Meeting**, Manchester, UK (Dr Brian Beagley, UMIST).

23-28 May 1993 **American Crystallographic Assoc. Meeting**, Albuquerque, NM, USA (Dr A. Larson, LANSCE H805, Los Alamos Nat'l Laboratory, Los Alamos, NM 87455, USA).

18-26 July 1993 **10th International Clay Conference**, Adelaide. (10th ICC Secretariat : Ellisservice Convention Management, PQB 753 Norwood, South Australia, Australia 5067).

21-29 Aug. 1993 **XVth IUCr General Assembly and International Congress of Crystallography**, Beijing, China. (Prof. M.C. Shao, Inst. of Physical Chemistry, Dept. of Chemistry, Peking U., Beijing 100871, China).

MAILING LIST FOR NEWSLETTERS

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CALL FOR CONTRIBUTIONS TO THE CPD NEWSLETTERS

The next issue of the CPD Newsletter will be edited by Dr D. E. Cox to appear in the autumn of 1992. He would greatly appreciate contributions from readers on matters of interest to the powder diffraction community, e.g. meeting reports, future meetings, developments in instruments, techniques, computer programs and news of general interest. Please send articles and suggestions directly to him at his address given below.

Many thanks,
D. Louër, Editor of this Newsletter

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