

# COMMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

NEWSLETTER No 2

December 1988

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## SOME OBSERVATIONS ON RECENT DEVELOPMENTS IN POLYCRYSTALLINE DIFFRACTION

This is a very exciting time in the field of powder diffraction. This note presents some impressions of what has occurred in the last decade to transform the powder method from a mature analytical technique to a rapidly expanding and much more powerful method with many new uses and users. Several important developments came to fruition in roughly the same time frame, including the ever-expanding power and availability of computers, the profile fitting method which has greatly improved data reduction and interpretation, the Rietveld method which has become an important method for crystal structure analysis, major advances in X-ray detectors, and the use of synchrotron radiation. There were also many innovations in commercial instrumentation, particularly the rotating anode generators, but there is not sufficient space to discuss them.

An example of the important uses of the powder method is the recent discovery of the high-temperature superconductors. This involved thousands of scientists and was accompanied by world-wide publicity. The importance of the method was demonstrated to scientists who had little or no previous interest in diffraction.

It immediately became the standard method to follow the progress of the solid state reactions by identifying the phases produced in the synthesis of the materials. The scientific race suddenly required powder laboratories to greatly expand their output and shorten the turn-around time. It was not unusual to operate the equipment continually and to sleep in the laboratory. Huge numbers of patterns were produced with physicists peering over the shoulders of the diffractionists to find if they had made a successful run. More advanced studies using X-ray and neutrons were made to determine the crystal structure and the site occupancy. This is a shining moment in the history of the powder method.

Profile fitting is used to determine the shape, scattering angle and width of each reflection, or a cluster of overlapping reflections, with much higher precision than it was possible with the analogue strip-chart recorder method. A computer is used to fit the step-scanned data with a Lorenz, pseudo-Voigt or other appropriate curve-fitting function. The instrument function inherent in the diffractometer geometry can be precisely determined and the broadening and peak shifts from the specimen can be analysed. Computer graphics provides a powerful direct means of exactly comparing the observed and calculated profiles. Subtle differences in shapes become apparent and are invaluable in determining particle size, microstrain and disorder stacking.

The Rietveld method has been used for a large number of structural studies of powder samples by neutron diffraction, and it is now being used for synchrotron X-ray powder data. Combining the results of neutron and X-ray diffraction and supplemented with electron microscopy when applicable will lead to better understanding of the complex materials now being developed.

Synchrotron radiation provides a powerful new X-ray source whose properties have made possible the development of improved and new powder methods. The high intensity, parallel beam and wavelength selectively are ideal for diffraction. Time-resolved analyses with position sensitive detectors are being developed. The systematic errors inherent in the conventional focusing methods are absent in the parallel beam geometry thereby, increasing the precision of lattice parameter determination. Each reflection is a single peak  $0.02^\circ$  to  $0.05^\circ$  wide and difficulties arising from the  $K\alpha$  doublet are absent. The wavelength can be selected to obtain the highest possible peak-to-background. Anomalous scattering can now be conveniently studied.

The parallel beam optics does not require the  $\theta - 2\theta$  specimen-detector relation. This makes possible thin film analysis by depth profiling to add another dimension to the characterization. The beam enters the film at grazing incidence — less than the critical angle — and only the detector is scanned. Only the top 50 to 200 Å of the film is penetrated, and the diffraction patterns from the thin surface can be compared to those of the full film thickness obtained with  $\theta - 2\theta$  scans. The preferred orientation, inclination of selected lattice planes to the film surface and other characteristics can be determined in addition to the phases.

Synchrotron radiation is well-suited to energy dispersive diffraction because of the high intensity continuous radiation. The resolution can be increased by a factor of  $10^2$  to  $10^3$  over conventional EDD by step scanning the incident beam monochromator so that the X-ray optics rather than the detector determines the resolution; scintillation counters can be used in place of solid state detectors.

The expanded activity is shown by the increasing numbers of papers involving the use of the powder method presented at scientific meetings. The new journal Powder Diffraction began publication in 1986 and supplements the Journal of Applied Crystallography and Advanced in X-Ray Analysis. The momentum generated is likely to continue and we may confidently look forward to greater use of powder diffraction.

W. Parrish



William Parrish

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## CALL FOR CONTRIBUTIONS TO THE COMMISSION AND ITS NEWSLETTER

Members of the powder diffraction community are invited to contact any member of the Commission on Powder Diffraction with matters for possible consideration by the Commission and/or inclusion

in subsequent (biannual) Newsletters. A matter for which input from the diffraction community is certainly needed relates to the selection (if deemed desirable) of a logo for the Commission.

Z. Bojarski  
Editor, Newsletter No 2

## RIETVELD REFINEMENT ROUND ROBIN

The CPD is undertaking a Round Robin Survey of the Rietveld Method for powder diffraction analysis. The first stage of this survey will involve

- (i) an intercomparison of crystal structure (including B values) and unit cell parameters, and their associated estimated standard deviations, obtained by Rietveld analysis of X-ray and neutron data collected on two standard powder samples, and
- (ii) an intercomparison of the various Rietveld analysis programs in common use.

The participating laboratories will be asked to collect either neutron or X-ray diffraction data on both samples, to obtain estimates of the structural parameters from this data with their in-house programs, and to submit both the raw data and the analytical results to the CPD. The CPD will then compare and contrast the submitted results for each sample with each other, and with the results obtained by reanalysis of the raw data using one or more "standard" Rietveld programs selected by the CPD.

The second stage of the Round Robin Survey will be initiated after the first stage is completed. It will involve an analysis of the more subtle aspects of Rietveld structure refinement, namely, the determination of crystal size and strain parameters, the treatment of preferred orientation, multiphase analysis, peak shape algorithms etc. Different samples will be used for this stage.

It is the intention of the CPD to distribute the two samples for the first stage of the survey before and/or during the International Workshop on the Rietveld Method to be held in Petten, the Netherlands, in June 1989. At this time, a CPD subcommittee appointed to manage the survey

will discuss details of the data collection and analysis procedures with the participants. The results of the survey will be presented during the Satellite Meeting on Powder Diffraction Analysis associated with the 15th Congress of the IUCr in Bordeaux, France, in July 1990, and will ultimately appear in published form.

If any reader wishes to participate in this survey, please fill out the data form below and send it to Dr. R. J. Hill at the address provided at the end of the form:

Name: .....

Institution: .....

Address: .....

Brief description of the available equipment(conditions) codes that would be used for survey. For example:

- (i) X-rays: conventional (synchrotron) **counter/film-scanner/energy** dispersive
- (ii) neutron: steady state/time of flight
- (iii) geometry: focusing/ parallel/flat specimen/spindle
- (iv) monochromation: filtered/incident-beam mon./diffracted-beam mon.
- (v) data collection: wavelengths/two-theta limit/minimum step size
- (vi) computer programs: origin/local modifications/special features

Address for sending replies: Dr. R. J. Hill  
Mineralogical Institute  
Univ. Würzburg, **Am** Hubland  
D-8700 Würzburg,  
FRG

R.J. Hill

## MEETING REPORT

### XIIITH CONFERENCE ON APPLIED CRYSTALLOGRAPHY

Cieszyn, Poland, August **23-27,1988**



Conference venue: Applied Arts Department, the Silesian University

Conferences on Applied Crystallography, organised regularly every two years since **1962** by Professor Bojarski and his colleagues from the Silesian University (Katowice) and the Institute of Ferrous Metallurgy (Gliwice), have long ago gained an enviable international reputation. The broad spectrum of subjects covered, the generally high standard of

the papers and their convenient presentation in two sessions (oral and poster), very pleasant living conditions and tourist facilities in an interesting locality, plus the very moderate costs, have made this Conference a popular forum for meetings and exchange of views and experience between scientists from the East and the West.

**TWELFTH EUROPEAN CRYSTALLOGRAPHY MEETING**  
Moscow, USSR, August 20 – 29, 1989

The conference will include invited lectures as well as micro-symposia and poster sessions. The main topics are:

\* fundamental crystallography, the theory of symmetry, computing methods \* experimental techniques and equipment for structural analysis of crystals \*

Planned postsymposia (August 27 – 28) include:

1. Practical aspects of protein crystallography
2. Synchrotron radiation in crystallography
3. Structure of partially ordered systems
4. Chemical bonding and lattice dynamics by diffraction methods

Please address all correspondence to:

Dr E. H. Harutyunyan  
XII European Crystallographic Meeting  
Institute of Crystallography  
Academy of Science of the USSR  
Leninsky pr. 59  
MOSCOW 117333, USSR

**12 INTERNATIONAL CONGRESS  
ON X-RAY OPTICS AND MICROANALYSIS**  
Kraków, Poland, August 28 – September 1, 1989

12 ICXOM is being organized by the Institute of Metallurgy, Academy of Mining and Metallurgy in Kraków, Poland. The scientific program may include the following topics:

\* quantitative analysis \* X-ray transmission analytical microscopy \* analytical scanning electron microscopy (electron emission and contrast) \* X-ray spectrometry \* electron optics \* signal and image processing \* secondary ion emission microscopy and microanalysis \* electron energy loss spectrometry \* other analytical techniques \* application in biology \* application to materials \*

In addition an exhibition of analytical instruments, analytical materials, reagents etc, books and Journals will be arranged at the conference.

Please address all enquiries and correspondence to:

12 ICXOM  
Institute of Metallurgy  
Academy of Mining & Metallurgy  
al. Mickiewicza 30  
PL – 30-059 KRAKOW, Poland  
Prof. S. Jasieniska

**XIII HUNGARIAN DIFFRACTION CONFERENCE**  
Balatonfüred, Hungary, May 21 – 25, 1989

The conference is organized by the Diffraction Group of the R. Eötvös Physical Society and should give a good chance for scientists coming from Hungary and other countries to 'present their latest scientific achievements and discuss their results on an international platform. HDC-89 will be held at Balatonfüred which is one of the most beautiful places at lake Balaton.

HDC-89 should cover three major topics:

- (i) New developments and application in powder diffraction, including texture and real structure studies
- (ii) Surface and defect characterisation by X-ray, neutron and electron diffraction methods
- (iii) The reliability of structure determination by X-ray, neutron and electron diffraction

For further information please contact:

Secretary of HDC-89  
Roland Eötvös Physical Society  
P.O. Box 433  
H – 1371 BUDAPEST, HUNGARY

**XIV CONFERENCE ON APPLIED CRYSTALLOGRAPHY**  
Silesian University, Cieszyn, Poland, August 1990

For information please contact:

Dr E. Lagiewka  
Uniwersytet Śląski  
Instytut Fizyki i Chemii Metali  
ul. Bankowa 12  
40-007 KATOWICE, Poland

**NEWS FROM OTHER IUCr COMMISSIONS**

The Commission on Powder Diffraction plans to keep a strong liaison with the Commissions on Crystallographic Apparatus, Neutron Diffraction, and Electron Diffraction because they have interest and responsibilities most closely allied to those of the CPD. In line with this policy, a few items of interest to the readers of the CPD Newsletter have been extracted from two recent newsletters of the Commission on Neutron Diffraction (CND).

**August 1986**

At the Argonne National Laboratory, the powder diffractometers continue to perform a great deal of new science. The ancillary equipment now includes high temperature (1500°C) furnaces into which special atmospheres such as CO/CO<sub>2</sub> mixtures can be placed, and high pressure cells. Multiphase codes and special peak-broadening routines are also available as part of the software package. A short course on powder diffraction, with particular emphasis on potential industrial applications, was held in May 1986 and the attendees were given the opportunity of hands-on experience.

**Commercial Neutron Diffraction at Chalk River**

The exploitation of neutron diffraction techniques to measure internal strains in manufactured components is accelerating rapidly at Chalk River. Drs. T. M. Holden, B. M. Powell and S. R. MacEwen have been remarkably successful in getting industry interested in the ability of neutrons to detect small changes in lattice parameters (a few parts in 10<sup>4</sup>) in a manner not achievable by any other technique. The penetrating power of neutrons enables them to probe internal strains, millimeter by millimeter, through bulky items like steel pipelines and Zircaloy pressure tubes.

**May 1988**

**Industrial Applications of Neutron Diffraction**

A 15-page Newsletter, edited by B. M. Powell, highlights the contributions from 22 laboratories as reported by 51 authors. The predominant interest pertains to small angle scattering, strain measurements and texture measurement on large samples, and powder diffraction analysis. (The large field of neutron radiography was deliberately excluded from this review.) "Industrial applications" range from scientific investigations of materials on potential industrial interest to full-scale commercial contract work of a proprietary nature. For a copy of this newsletter write to Dr. Sax Mason, Institut Laue-Langevin, BP 155X, 38042 Grenoble Cedex, France.

**Members of the Commission Neutron Diffraction:**

A. Albinati	Italy
M. Iizumi	Japan
B. Lebech	Denmark
S. Mason	France
B. M. Powell	Canada
W. Prandl	W. Germany
A. S. Sequeira	India
H. G. Smith	USA
B. T. M. Willis	England
C. T. Ye	China

L. K. Frevel

## ACTIVITIES OF THE POLISH POWDER DIFFRACTION GROUP

The Polish Powder Diffraction Group has been in existence since 1960, initially on a non-formal footing, then from 1966 as the Applied Crystallography Commission, forming part of the National Crystallography Committee, affiliated to the IUCr. The activities of the Group, and next Commission, have been directed since its inception by Professor Bojarski of the Silesian University in Katowice.

The interests of the Powder Diffraction Group are focused principally on promoting the fullest utilisation of data obtained from detailed analysis of powder diffractograms to ascertain the characteristic properties of materials. This envisage: qualitative and quantitative phase analysis, precision measurements of crystal structure, analysis of lattice parameter defects, investigations of texture, size of crystallites and stresses.

The activities of the Applied Crystallography Commission cover broad spectrum of methods for investigation of materials, dominating here are the methods using scattering of X-rays, electrons and neutrons on polycrystalline materials. The Commission also deals with the micro-diffraction method (**Kossel**) and analysis of chemical composition in microareas (X-ray microanalysis).

The materials studied in the Commission's methodological investigations include metals, minerals, ceramic materials, plastic, fibres. Special attention is paid to research on phase transitions, particularly those finding applications in the development of new technologies.

The main objective of these activities is to speed up and propagate new research methods, facilitating and initiating contacts between scientists and promoting activities in Polish research centres. These objectives are achieved by the organisation of regular international „Conferences on Applied Crystallography” (already 13 conferences have been held), summer Schools on selected research topics, seminars on X-ray microanalysis, research testing, interview-questionnaires covering all the Polish scientific centres applying powder diffraction methods, preparing papers and publications on advances made in specific research areas.

The Commission maintains contacts with all the Polish and also foreign scientific centres making use of applied crystallographic methods. The Commission also takes an active part in work initiated by the National Crystallographic Committee.

Address: Applied Crystallography Commission  
Professor Zbigniew Bojarski  
Institute of Physics and Chemistry of Metals  
ul. Bankowa 12  
40-007 Katowice, Poland

T. Bold

## FORTHCOMING MEETINGS OF INTEREST TO POWDER DIFFRACTIONISTS

### WORKSHOP ON RIETVELD ANALYSIS:

NERF, Petten, The Netherlands, June 13 – 15, 1989

The meeting is organized under the auspices of the IUCr Commission on Powder Diffraction. The ECN have responded very enthusiastically to the selection of them as the host institution. Dr. C. van Dijk has accepted the chairmanship of the Local Committee and Dr. A. Hewat has accepted the chairmanship of the program committee. The IUCr have provided some money for assistance to young scientists wishing to attend the Workshop. Selection criteria for these competitive grant awards will be based on demonstrated promise and the degree to which an award will truly be helpful to the applicant's career. The amount of each award will be based on actual economy travel costs, other sources of support available, etc., and will not exceed US \$ 500 each. Application forms will be available by 1 January 1989 from:

Dr. Cor van Dijk, Chairman  
ECN, P. O. Box 1,  
1755 ZG Petten,  
The Netherlands

or from the CPD chairman

Prof. R. A. Young  
School of Physics,  
Georgia Institute of Technology,  
Atlanta, Ga, 30332, USA

### XVTH INTERNATIONAL CONGRESS AND GENERAL ASSEMBLY OF THE IUCr

Bordeaux, France, July 19 – 28, 1990

The Congress will be held on the Campus of Bordeaux University. It will include invited general lectures, microsymposia and poster sessions. Some microsymposia will consist in open Commission Meetings. In addition discussion sessions following poster sessions will be organized experimentally in a certain number of cases. All National Committees and all Commissions of the Union are invited to submit proposals for all part of the scientific program. The suggestions should be sent by February 28th, 1989 to Professor A. Authier, Chairman of the Program Committee, Laboratoire de Mineralogie et Cristallographie, Université P et M Curie, 75252 Paris CEDEX 05.

Some CPD members having discussed the problem at ECM-11 in Vienna recommend that the powder diffraction session topic(s) and the SM-90 program topics be selected in concern so that the two programs complement each other and so that the SM-90 will appeal to powder diffraction specialists while the Bordeaux powder session(s) will appeal to a more general audience. They offer the following examples of possible topics that could each be the subject of a half-day session:

- (i) *ab initio* structure determination from powder diffraction data
- (ii) complementarity and contrasts in the use of synchrotron – X-ray and of neutron powder diffraction

For further information please contact:

Dr. M. Hospital,  
Laboratoire de Cristallographie  
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33405 TALECE CEDEX, FRANCE

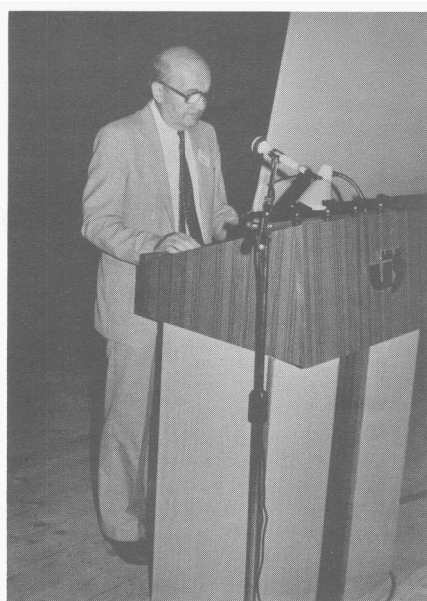
### SATELLITE MEETING ON POWDER DIFFRACTION

Toulouse, France, July 16 – 19, 1990

In connection with the XV International Congress of Crystallography in Bordeaux, France, the CPD are organizing a Satellite Meeting on Powder Diffraction to be held in Toulouse. The Chairman of the Local Organizing Committee is Dr. Jean Galy, long-time head of a well known group of crystallographers now seriously taking up powder diffraction.

The program committee for the meeting has not yet been selected but it is agreed that the program should include many aspects of powder diffraction while still having programmatic coherence, should emphasize things other than Rietveld refinement, and should complement and not compete with the main congress. Some suggestions for the program are as follows:

- Instrumentation and high resolution powder diffraction
- Microstructural characterization
- Total pattern fitting
- *Ab-initio* structure determination
- Specific applications
- Quantitative analysis and phase identification.



Prof. Z. Bojarski during his speech at the opening ceremony

diffraction, the use of synchrotron radiation in topography, new analytical methods for texture analysis, precision methods for measurement of lattice parameters and many others. Considerable interest was aroused by the paper read by Professor R. A. Young on "Crystallite size, microstrain indicators in Rietveld refinement" and that given by Professor D. K. Smith on "The use of the full diffraction trace, both experimental and calculated in quantitative X-ray powder analysis". A popular attraction at this Conference was the seminar Workshop on JCPDS, led by Professor D.K. Smith and Dr J. Visser, where a discussion took place on both the elements of phase identification and the newest computer programs. All the papers presented at the Conference were published, in English, as "Proceedings of the XIII Conference on Applied Crystallography" (567 pages). Those interested in obtaining a copy of Proceedings (price US \$ 50) are invited to contact the organisers:

Institute of Physics and Chemistry of Metals  
Silesian University  
Bankowa 12  
40-007 Katowice, Poland

A certain number of copies of Proceedings from previous Conferences are still available.

We may confidently anticipate that the next Conference will successfully be arranged to take place during the summer vacation period in 1990.

E. Lagiewka



The most recent, XIIIth Conference on Applied Crystallography was held from 23 – 27th August in Cieszyn, an ancient historic town on Poland's southern border, where the Silesian University has one of its departments – the Applied Arts Department. The timing of the Conference was chosen so that at its close the participants could conveniently travel direct to the XI European Crystallography Meeting (Vienna, 28.08 – 2.09).

Of the total number of persons taking part almost half were foreign participants from thirteen countries: USSR, Czechoslovakia, German Democratic Republic, Bulgaria, Holland, Hungary, Italy, China, France, Greece, Japan, Turkey and USA. The most numerous group, apart from the Polish participants, was formed by Soviet scientists.

Altogether 123 papers were submitted to the Conference, of which 27 were given at plenary sessions and 74 at poster sessions. Unfortunately, a certain number of papers received were not presented due to the non-appearance of the authors. All the papers were presented in English. The subject areas covered were the utilisation of crystallographic methods for investigations on metal alloys, ceramic and glass materials, polymers, minerals etc. In research on metals interest was focused primarily on phase transitions and in particular the martensitic transformation, the amorphous structure of metal alloys in the initial stages of their crystallisation, texture analysis of plastically deformed and recrystallised metals, description of the dislocation structure of single crystals employing topographical methods etc. Studies on metals clearly dominated over crystallographic applications in other groups of materials. It may be claimed that researches on the martensitic transformation, particularly on shape memory alloys and analysis of texture in metals has become the speciality of the Polish participants in the Conference.

Another group of papers, also numerous, dealt with developments in applied crystallography methods, in this group papers were presented on methodology in phase identification, phase transitions, small-angle scattering, a new conception of radial distribution of intensity, identification of weak reflections in the background, the application of electron



At the get-together party

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## FUTURE ACTIVITIES OF THE COMMISSION ON POWDER DIFFRACTION

The International Workshop on the Rietveld Method and the Round Robin on Rietveld refinement as well as the Satellite Meeting on Powder Diffraction in Toulouse are treated elsewhere in this Newsletter.

The CPD will also offer to organize one or more sessions **at** the main Congress in Bordeaux. One theme being seriously considered is **ab initio** structure determination from powder data.

The program Exchange Bank project mentioned in Newsletter No. 1 is developing slowly. The final form is not yet clear; it may be simply an information exchange, or perhaps a collaboration with

another group who actually bank and provide programs, or something else not yet envisioned. These questions should be resolved by **the** time of the Bordeaux Congress.

The possibility of organizing in Eastern Europe, perhaps Poland, a Summer School for Beginners with the Rietveld Method is being investigated.

Proposed issue dates and (editors) for the next few Newsletters are July 1989 (Dr. Yamanaka), Dec 89 — Jan90 (Prof. Werner), June **1990** (Dr. Langford), and Dec 1990 (Dr. Hewat or Prof. Young).

R. A. Young

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## MAILING LIST FOR FUTURE NEWSLETTERS

If you wish to receive a copy of future CPD Newsletters please complete the following coupon, or its copy, and return to Dr Langford at the address below:

To the IUCr Commission on Powder Diffraction:

\_\_\_\_\_ Please keep me on the mailing list for future issues of the CPD Newsletter.

\_\_\_\_\_ Please add the following interested person to your mailing list.

Name: \_\_\_\_\_

Position/Title: \_\_\_\_\_

Address: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Return this form, filled out, to:

Dr J. I. Langford  
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