IUCr COMMISSION NEWS

Contributions intended for this section should be submitted to The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

COMMISSION ON POWDER DIFFRACTION

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Newsletter No. 2 (Extracts)

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 high-temperature superconductors. This involved thousands of scientists and was accompanied by worldwide 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-rays 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 analog strip-chart recorder method. A computer is used to fit the step-scanned data with a Lorentz, 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 analyzed. 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, 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 selectivity 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 to 0.05° wide and difficulties arising from the Kα doublet are absent. The wavelength can be selected to obtain the highest possible peak-to-background ratio. Anomalous scattering can now be conveniently studied.

The parallel-beam optics does not require the θ–2θ 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 with those of the full film thickness obtained with θ–2θ 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 (EDD) 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 *Advances 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

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 these 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 a copy of the following data form and send it to Dr R. J. Hill at the address provided below:

Name: .....

Institution: .....

Address: .....

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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/diffracted-beam;

(v) data collection: wavelengths/2θ limit/minimum step size;

(vi) computer programs: origin/local modifications/special features.

Address for replies:

Dr R. J. Hill
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R. J. Hill

XIIIth Conference on Applied Crystallography, Cieszyn, Poland, 23–27 August 1988

Conferences on Applied Crystallography, organized regularly every two years since 1962 by Professor Bojarski and his colleagues from the Silesian University (Katowice) and the Institute of Ferrous Metallurgy (Gliwice), 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 experiences between scientists from the East and West.

The XIIIth Conference on Applied Crystallography was held from 23 to 27 August 1988 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 XIth European Crystallographic Meeting (Vienna, 28 August–2 September 1988).

Almost half of the participants were foreign from 13 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, some of the papers received were not presented due to the nonappearance of the authors. All the papers were presented in English. The subject areas covered were the utilization 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 crystallization, texture analysis of plastically deformed and recrystallized metals, description of the dislocation structure of single crystals employing topographical methods *etc.* Studies on metals clearly dominated crystallographic applications in other groups of materials. It may be claimed that research on the martensitic transformation, particularly on shape-memory alloys and analysis of texture in metals, has become a speciality of the Polish participants at 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 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 XIIIth Conference on Applied Crystallography' (567 pp.). Those interested in obtaining a copy of the proceedings (price US $50) are invited to contact the organizers:

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

A certain number of copies of the proceedings of previous conferences are still available.

We may confidently anticipate that the next conference will successfully be arranged for the summer vacation period in 1990.

E. Łagiewka

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 later 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 utilization of data obtained from detailed

analysis of powder diffractograms to ascertain the characteristic properties of materials and include 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 a broad spectrum of methods for investigation of materials, particularly methods invoving the scattering of X-rays, electrons and neutrons by polycrystalline materials. The Commission also deals with the microdiffraction method (Kossel) and analysis of chemical composition in micro-areas (X-ray microanalysis).

The materials studied in the Commission's methodological investigations include metals, ceramic materials, plastics and 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 organization of regular international 'Conferences on Applied Crystallography' (13 conferences have already been held), summer schools on selected research topics, seminars on X-ray microanalysis, research testing, interview questionnaires covering all the Polish scientific centres using 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. Its address is:

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40-007 Katowice, Poland

T. Bołd

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 Symposium on Powder Diffraction in Toulouse are treated elsewhere in the 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 on the Rietveld method is being investigated.

Proposed issue dates (and editors) for the next few newsletters are July 1989 (Dr Yamanaka), December 1989–January 1990 (Professor Werner), June 1990 (Dr Langford), and December 1990 (Dr Hewat or Professor Young).

R. A. Young

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.

Z. Bojarski
Editor, Newsletter No. 2

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