Book Reviews

Works intended for notice in this column should be sent direct to the Book-Review Editor (R. F. Bryan, Department of Chemistry, University of Virginia, McCormick Road, Charlottesville, Virginia 22901, USA). As far as practicable, books will be reviewed in a country different from that of publication.

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Electron diffraction techniques. Vol. 2. (**IUCr Monographs on Crystallography** No. 4.) Edited by JOHN M. COWLEY. Pp. vi + 423. Oxford: Oxford University Press, 1993. Price £45.00. ISBN 0-19-855733-7.

This is the second of two volumes, sponsored by the Commission on Electron Diffraction of the International Union of Crystallography, that are intended to be a comprehensive overview, written by acknowledged experts, of electron diffraction theory and its application to materials problems. A review of Volume 1 has already appeared [*Acta Cryst.* (1993), A**49**, 677-678].

Over half of Volume 2 is the detailed Chapter 1 (222 pp.) on the analysis of defects by diffraction contrast and high-resolution electron microscopy, written by S. Amelinckx and D. Van Dyck of the University of Antwerp. This, in itself, could very well have been published as a separate monograph. These authors also have a second contribution to the volume, Chapter 4 (65 pp.), which is a very interesting treatment of electron diffraction from modulated structures. Other contributions include Chapter 2 (37 pp.) by J. K. Gjønnes of the University of Oslo, which discusses the analysis, in electron diffraction patterns, of continuous diffuse scattering caused by crystal disorder or thermal motion. Chapter 3 (49 pp.), by K. Yagi of the Tokyo Institute of Technology, is a description of reflection high-energy electron diffraction and reflection electron-microscopy techniques for the examination of crystal surfaces. Finally, the use of electron diffraction patterns and spectroscopic techniques (primarily EDS) to identify unknown materials is treated in Chapter 5 (45 pp.), by C. E. Lyman (Lehigh University) and M. J. Carr (Sandia National Laboratories), expanding the rather terse treatment of this topic in Volume 1.

For the investigator interested in inorganic materials, there is much to praise in this volume, as there was also in the preceding one. The theoretical treatments in Chapters 1 and 4, in particular, are quite thorough and will serve as a handy reference to researchers interested in the analysis of real crystal structures, including defects, disorder and deviations from simple stoichiometry. [It is a shame, however, that three important figures (1.4, 1.6 and 1.7) are missing.] Chapter 2 gives a balanced overview of how diffuse scattering problems have been analyzed and Chapter 3 is most fascinating in its treatment of surface dynamic processes. The difficulties of using crystallographic databases with analytical electron diffraction data are clearly portrayed by the authors of Chapter 5, who, nevertheless, give practical advice on how unknowns can be identified with such data. There is much in this volume that would be of use to the organic materials scientist also - especially since the advent of low-dose electron imaging techniques has made possible the analysis of defects in polymer crystals, for example, in terms of molecular (if not atomic) packing. Personally, I was most interested in the chapter on incommensurate structures because, after I read it, it became clear to me that this development could also be applied to the analysis of the phase separation of metastable paraffin solid solutions. Hence, as ever, the organic materials scientist follows the harbingers in inorganic materials research. Obviously the harbingers are not so much troubled by electron-beam-induced radiation damage as are the followers!

Excellent as the contributions to Volumes 1 and 2 of this series are, the reader should be cautioned that the predominant viewpoint of most authors therein is that no direct analysis of the experimental electron diffraction or microscopic data is possible and that only an indirect approach, starting from an assumed crystal structure model, can be used. This, of course, is a result of the complexity of the dynamical scattering theory. Even with an indirect interpretation of these data by image simulation, the viewpoint can also be quite negative. To quote a passage from Chapter 1: 'Nevertheless, the technique is so tedious and so insensitive that, thus far, it has been only successful if the number of plausible structure models is very limited. In our view it is astonishing that such an expensive technique as HREM (high-resolution electron microscopy) is so dependent on the availability of prior information obtained from other techniques'.

It is important, therefore, to point out that there are other, more optimistic, opinions. For example, a review of high-resolution electron microscopy in solid-state chemistry by L. Kihlborg of Stockholm University [*Prog. Solid State Chem.* (1990), **20**, 101-133] demonstrates that a direct structure analysis is sometimes permitted, even with inorganic materials. This is exemplified by the numerous studies by S. Hovmöller and his collaborators [*e.g. Nature* (*London*) (1984), **311**, 238-241, and many more recent papers]. The chapter on modulated structures was probably written before the publication of the ingenious analysis of ankangite by Xiang, Fan, Wu, Li & Pan [*Acta Cryst.* (1990), A**46**, 929-934], which describes the application of direct phasing methods to electron diffraction intensity data. Similar work has followed since then, such as that from the laboratory of Fan Hai-fu and Li Fang-hua. These studies do not state, by any means, that the scattering theory is incorrect, but only that this theory can be exploited for collection of data sufficiently near the single-scattering approximation to permit a direct structure analysis to be carried out. Such analyses, moreover, can yield chemically reasonable results in agreement with (but not dependent upon) independent X-ray crystallographic studies. This has been recognized by organic electron crystallographers for years.

With these reservations in mind and with continued regrets that the organic field is not more fully represented (for instance by proteins or linear polymers, each with a long history of electron crystallographic structure determinations), there are no doubts in this reviewer's mind that these two volumes represent a significant addition to any electron diffraction-

ist's bookshelf, with extensive material available for study in considering new research directions.

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Modern powder diffraction. Reviews in mineralogy, Vol. 20. Edited by D. L. BISH and J. E. POST. Pp. xi + 369. Washington, DC: The Mineralogical Society of America, 1990. Price (paper) US $25.00. ISBN 0-939950-24-3.

At the November 1989 annual meetings of the Mineralogical Society of America and the Geological Society of America in St. Louis, D. L. Bish and J. E. Post convened a short course on advanced methods in powder diffractometry. The course content has been published as one of the *Reviews in Mineralogy* series familiar to subscribers to *American Mineralogist*.

Powder diffraction is currently one of the most exciting subfields of crystallography. Recent advances in light sources, instrumentation and software have greatly increased the amount of information available from a powder diffractogram. The authors seek to illustrate the state of the art in powder diffraction, to describe how to obtain high-quality powder diffraction data and to demonstrate how to extract maximum information from the data.

This volume consists of 11 chapters by several prominent authors. In *Principles of powder diffraction*, R. C. Reynolds gives a brief summary of the principles of powder diffraction and basic diffraction theory. This treatment should be useful for beginners but is also worthwhile for experts to review occasionally. Ron Jenkins discusses *Instrumentation* and *Experimental procedures* well in limited space. The discussion of instrumentation neglects to illustrate Guinier geometry and to discuss the special problems associated with synchrotron radiation but is a useful refresher. The treatment of experimental procedure highlights the variety of powder diffraction instruments, applications and users. In *Sample preparation for X-ray diffraction*, D. L. Bish and R. C. Reynolds give a short and accurate discussion of grinding and (underappreciated) particle size effects. Understanding these effects is critical for obtaining high-quality data, especially at synchrotron sources. This discussion duplicates some of the material in Chapter 3. Especially useful is the treatment of microabsorption on pp. 82–83. Also discussed are specialized clay sample-preparation techniques.

The treatment of *Quantitative analysis* by R. L. Snyder and D. L. Bish summarizes classical methods, pointing out the potential microabsorption effects on internal standard methods. The exciting developments in using Rietveld analysis for quantitative analysis are demonstrated, as is D. K. Smith's development of quantitative analysis techniques using observed patterns. This last technique is particularly useful for amorphous and poorly crystalline materials, as well as for phases of unknown structures. Chapter 6, *Diffraction by small and disordered crystals* is, in contrast to the rest of the book, a specialist discussion of the diffraction encountered in clays and other low-dimensional materials. D. K. Smith summarizes the contents of the various crystallographic databases and discusses the software available for performing various powder diffraction tasks in *Computer analysis of diffraction data*.

The core of the book lies in Chapter 8, *Profile fitting of powder diffraction patterns*, by S. A. Howard and K. D. Preston, and in Chapter 9, *Rietveld refinement of crystal structures using powder X-ray diffraction data*, by J. E. Post and D. L. Bish. These chapters are an excellent summary of the mathematics and history of profile fitting, complete with discussion of the pitfalls and sources of error. The authors summarize coherently things that many of us have learned the hard way! A cogent discussion of the trade-offs in data collection and refinement strategy is given. Particularly useful are the discussions of errors, false minima and preferred orientation. The listing of normalized profile functions on p. 218 will be consulted often. There are hints of the power of difference Fourier techniques when applied with care to powder data.

The characteristics of synchrotron radiation and its applications are summarized well by L. W. Finger in *Synchrotron powder diffraction*. It is very useful to have documentation of the pseudo-Voight profile function used in the generalized structure analysis system (GSAS) of A. Larson and R. B. Von Dreele. Although brief, the discussion of sample preparation for synchrotron experiments covers the key points. R. B. Von Dreele summarizes briefly the history of neutron diffraction, instrument design and applications - particularly hydrogen location and cation distribution. The potential power of combined neutron/X-ray studies is pointed out.

Although part of a series of reviews in mineralogy, this book belongs on the shelves of every powder diffractionist and, most importantly, on those of members of the new species, powder crystallographers. The book is well produced and there are very few errors. Better buy two - both of my copies are already getting dog-eared.

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Introduction to crystallography. (Revised edition.) By C. HAMMOND. Pp. x + 132. Oxford: Oxford University Press for the Royal Microscopical Society, 1992. Price £10.95 (paper). ISBN 0-19-856433.

This short text is designed as a 'user-friendly' handbook of elementary crystallography. As its sponsorship indicates, it is intended to introduce practising microscopists to those crystallographic concepts that are essential in understanding