Laboratory Note

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A technique for controlling preferred orientation in powder diffraction samples*

The technique for forming spherical agglomerates from solids suspended in a liquid (Sirianni, Capes & Puddington, 1969) can be used on a laboratory scale with normal laboratory apparatus to eliminate, in the cases we tested, preferred orientation in X-ray powder diffraction samples. Smith & Barrett (1979) in a recent review have described the problem of obtaining a randomly oriented sample as 'probably the most difficult problem in sample preparation' and summarized methods for obtaining such samples. Apparently, liquid-phase agglomeration has not been used for this purpose. In this process (see *e.g.* references in Capes, McIlhinney & Sirianni, 1977), finely divided solids in liquid suspension can be agglomerated and separated from the suspending liquid by the addition of a small amount of a second liquid which preferentially wets the solid and is immiscible with the first liquid. With a certain amount of bridging liquid and suitable agitation (as in a laboratory sample shaker or high-speed domestic blender) the solids are separated as highly spherical bodies of controllable size. This process has become known as 'spherical agglomeration'. With water as the suspending fluid and varsol as the binding liquid (any hydrocarbon immiscible with water would be suitable), we have tested this technique on platy materials (graphite, talc and palygorskite) and fibrous brucite $[Mg(OH)_2]$ from the Jeffery Mine, Quebec. Spheres ranging from 50 μm to 1 mm in diameter were produced. The size of the plates or fibres was reduced by grinding to considerably below that of the spheres desired. Transmission photographs of stationary specimens (either one large sphere or a few small spheres on a fibre mount) showed random orientation. As this technique uses ordinary laboratory apparatus and small amounts of material it should be of use to overcome problems of preferred orientation in powder specimens. For some materials the grinding required could result in line broadening which might be a limiting factor in some applications.

L. D. CALVERT
A. F. SIRIANNI

Chemistry Division
National Research Council of Canada
Ottawa
Ontario K1A 0R6
Canada

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Crystallographers

This section is intended to be a series of short paragraphs dealing with the activities of crystallographers, such as their changes of position, promotions, assumption of significant new duties, honours, etc. Items for inclusion, subject to the approval of the Editorial Board, should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 5 Abbey Square, Chester CH1 2 HU, England).

Professor **J. M. Cowley,** Department of Physics, Arizona State University, USA, and Professor **M. M. Woolfson,** Department of Physics, University of York, England, have resigned as Co-editors of *Acta Crystallographica*. They have been succeeded by Professor **R. Collela,** Physics Department, Purdue University, USA, and Dr **B. T. M. Willis,** Materials Physics Division, AERE Harwell, England. Dr **S. Jagner,** Department of Inorganic Chemistry, Chalmers University of Technology and University of Göteborg, Sweden, has also been appointed a Co-editor, whilst Dr **M. Hospital,** Laboratoire de Cristallographie et de Physique Cristalline du CNRS, Talence, France, was appointed a Co-editor earlier this year.

A special model-making unit, employing handicapped people, is to be set up in the Department of Chemistry of the University of Edinburgh. The unit will be known as the Beevers Miniature Model Unit in Honour of Dr **Arnold Beevers,** who was Reader in the Department until his retirement two years ago.

Professor **E. C. Frank,** Henry Overton Wills Emeritus Professor, H. H. Wills Physics Laboratory, Bristol, England, has been elected a foreign associate of the USA National Academy of Engineering.

Dr **O. Kennard,** University Chemical Laboratory, Cambridge University, England, has received the 1979 Chemical Society Award in Structural Chemistry for her work with particular reference to biologically important molecules.

Dr **R. A. Laudise,** Director of the Physical and Inorganic Chemistry Research Laboratory, Bell Telephone Laboratories, Murray Hill, New Jersey, has been elected a member of the USA National Academy of Engineering.

Professor **J. V. Smith,** Department of Geophysical Sciences, University of Chicago, USA, has been awarded the 1980 Murchison Medal of the Geological Society of London in recognition of his distinguished contribution to crystallography, mineralogy and petrology.

Book Reviews

Works intended for notice in this column should be sent direct to the Book-Review Editor (J. H. Robertson, School of Chemistry, University of Leeds, Leeds LS2 9JT, England). As far as practicable books will be reviewed in a country different from that of publication.

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Advances in X-ray analysis, Vol. 22. Edited by *G. J. McCarthy, C. S. Barrett, D. E. Leyden, J. B. Newkirk* and *C. O. Ruud.* Pp. xvii +492. Plenum Press, 1979. Price, US $ 42.50.

The articles in this volume, which is the proceedings of the 1978 Denver Conference, have been collected into seven sections: special techniques in powder diffraction; evaluation of XRD patterns; applications of XRD; instrumentation and laser sources; mathematical data analysis for XRF; XRF applications; XRF innovations.

In the first part of the book the authors consider problems in the analysis of materials by means of certain diffraction techniques, such as high- and low-temperature analysis, minimization of errors due to preferred orientation, and characterization of thin films, which allow the study of materials in the special conditions of employment.

In the second section, the principles of quantitative evaluation of the results of X-

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ray diffraction, by sophisticated computer methods, are collected.

The third section reports the results obtained by the application of X-ray diffraction to a variety of materials – from historical monuments to the identification of pigments and the study of tension states in metallic alloys. New alternative X-ray sources, particularly suitable for microradiography, are examined in the fourth section.

In the fifth, sixth and seventh sections, the problems of X-ray fluorescence (XRF) are considered. First the evaluation and interpretation of the results of XRF techniques are considered; then their application to particular materials, such as normal and stainless steels, is discussed; and finally, some new X-ray analysis systems are proposed, some using a single technique and others several different ones.

This text is therefore particularly interesting for users of X-ray diffraction and X-ray fluorescence because it offers a series of monographs of both theoretical and practical character. The book will be of value to the experts for the identification of new research fields in the applications of X-ray analysis.

M. GIORDANI
Istituto di Scienze e Tecnologie dell'Ingegneria Chimica
Universita di Genova
Via all'Opera Pia 11
16145 *Genova*
Italy

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Industrial crystallization 78 (Proceedings of the 7th Symposium on Industrial Crystallization,

held in Warsaw, Poland, September 1978). Edited by *E. J. De Jong* and *S. J. Jancic*. Pp. xiv + 588. Amsterdam: North Holland Publishing Company, 1979. Price US $ 73.25, Dfl 150.00.

The book consists of the following chapters: *Nucleation* (86 pp.), *Impurities and crystal growth* (119 pp.), *Hydrodynamics of crystallizers* (77 pp.), *Data measurement and crystallizer design* (106 pp.), *Industrial experience in crystalization* (99 pp.) and '*Poster session*' (79 pp.).

The chapter on nucleation contains nine papers: on precipitations (3), secondary nucleation (3), homogeneous/heterogeneous nucleation, Ostwald ripening, and a review. The chapter on impurities and crystal growth contains 11 papers; there are reports on the precipitation of a binary electrolyte, on potassium alum and sucrose, as well as on growth and growth dispersion, the growth of small crystals, the influence of collisions on the growth of crystals, the control of crystal habit, the effect of hydrodynamic conditions on crystal growth and generally on the role of impurities.

In the chapter on hydrodynamics of crystallizers (seven papers), the topics are: hydrodynamics and classification, the role of geometry, impeller power consumption, mixing intensity and contoured-base crystallizers. The chapter on data measurement and crystallizer design (ten papers) has contributions on kinetics, simulations and design theories, population models, batch fluidized beds, classification of crystallizers and crystallizers with quickly rotating horizontal cooling surfaces. The chapter on industrial experience in crystallization (11 papers) consists of contributions on carnellite, diammonium phosphate, sodium hydrogen carbonate, as well as on recrystallization through fluctuation, a direct-contact-type crystallizer with secondary refrigerant, a pelletizing process, classification, crystallizer selection and continuous centrifuges. The chapter reporting the poster session of the symposium (36 papers) is full of material. Here details are given for the following substances: potassium bromate surfactant solutions, colloid systems, gypsum, sucrose, ice, potash alum, sodium bicarbonate, calcium sulphate and ADP. Also discussed are: particle aggregation, precipitation, multistage countercurrent crystallization, fractional crystallization, periodic and stochastic temperature changes, temperature stabilization in liquid-jacket crystallizers, a scraped-surface heat exchanger, vacuum pans, vacuum crystallizers, incrustation in crystallizers, the system crystallizer/filter, and crystallization due to chemical reaction and salting out.

The book is well produced and contains numerous (281) very good diagrams, flow sheets and drawings of crystallizers. Any specialist will handle it with pleasure because it will give him much stimulation and it relates new results from the frontiers of the science. The individual papers are interesting and informative, with few exceptions. But, more and more, the abundance of material (over 80 papers and 617 references) squeezes even the specialist to death. Whilst formerly the chemical engineer could not obtain a tailor-made crystallizer design because only a little basic knowledge was available, today he is confronted with a lot of methods, which make his choices difficult or even risky. Therefore, these reviews and surveys have an increased importance. Each chapter contains at least one review. This is especially commendable; but a stronger summary of the individual papers, with a compilation of their comparisons and differences would have been a great help for the layman – and even for the specialist.

G. MATZ
Ingenieurabteilung AP
Verfahrenstechnik
Bayer AG
Friedrich Ebert Strasse 217/319
D 5600 *Wuppertal-Elberfeld*
Federal Republic of Germany

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Scanning electron microscopy/1979. Parts I and II.

Edited by *O. Johari*. Pp. part I: x + 598; part II: xviii + 910. Scanning Electron Microscopy, Inc., PO Box 66507, AMF O'Hare (Chicago), IL 60666, USA. Price, parts I and II together, US $ 131.00.

These volumes contain most of the papers presented at the Scanning Electron Microscopy 1979 conference which took place in Washington, USA, in April, 1979.

The conference was organized by SEM Inc., Dr Om Johari being the director of the meeting; the various parts were prepared by several University and Industry advisors.

The increasing interest of scanning electron microscopy (SEM) techniques in physical and biomedical sciences is revealed by the number of persons who attended the conference, and by the number of papers; this resulted in the publication of three volumes this year (of which only the first two are reviewed in the present article, as the third is devoted wholly to biomedical applications); from 1967 to 1977 only one volume was needed and in 1978 two volumes were required.

As for the past proceedings, the strongest point appears to be, after each paper, the publication of a reviewer's questions and the author's replies; these dialogues contain information essential to every SEM user.

Owing to the large range of subjects, the division of the papers is quite arbitary