

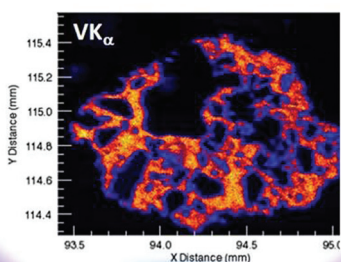
Powder Diffraction PDJ

JOURNAL OF MATERIALS CHARACTERIZATION

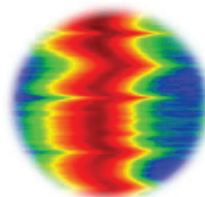
Proceedings of the Australian X-Ray Analytical Association (AXAA)
Conference held February 9–13, 2014

FROM MINERALS TO MATERIALS

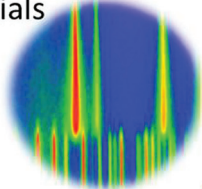
Complex Ores



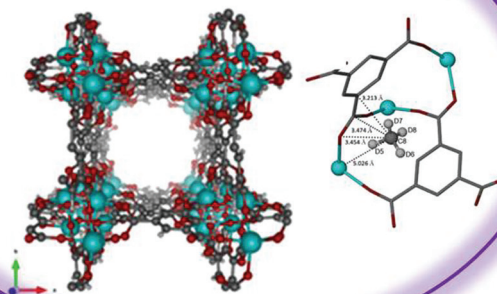
Battery Materials



Oxygen Storage
Materials



Porous Sorbents



LET OUR TEAM OF EXPERTS HELP YOU TAKE YOUR SKILLS TO THE NEXT LEVEL!

Practical X-ray Fluorescence: 27 April–1 May 2015

From theory to hands-on exercises, this course offers techniques and skills to improve lab performance. Discover the latest in cutting-edge instruments such as TXRF, hand-held devices, energy dispersive and wavelength dispersive spectrometers through live demonstrations.

The XRF course covers the basics of X-ray spectra, instrumentation design, methods of qualitative and quantitative analysis, specimen preparation and applications for both wavelength and energy dispersive spectrometry. The course emphasizes quantitative methods, use of automated X-ray spectrometers, review of mathematical matrix correction procedures and new developments in XRF. Submit your samples for analysis by the XRF experts. Selected results will be the basis for class discussion!

Fundamentals of X-ray Powder Diffraction: 1–5 June 2015

For the novice with some XRD knowledge or for the experienced with an interest in the theory behind XRD, this clinic offers a strong base for increased lab performance.

The clinic covers instrumentation, specimen preparation, data acquisition and qualitative phase analysis. Hands-on use of personal computers for demonstration of the latest software; data mining with the PDF. The powder diffractometer: optical arrangement, factors affecting instrumental profile width, choice and function of divergence slit, detectors, X-ray optics, calibration and alignment.

***Advanced Methods in X-ray Powder Diffraction: 8–12 June 2015**

For the experienced XRD scientist, this clinic offers enhanced analysis skills through intense problem solving, as well as an introduction to the Rietveld Method. Computer-based methods of data collection and interpretation, both for qualitative and quantitative phase analysis is also emphasized.

The advanced clinic covers factors affecting d-spacing of crystals: unit cell, crystal structure, and solid solutions, as well as factors affecting diffraction-line intensities: relative and absolute intensities, structure-sensitive properties (atomic scattering and structure factors), polarization effects, and multiplicity, specimen-sensitive effects (orientation, particle size), measurement-sensitive effects (use of peak heights and peak areas), and choice of scanning conditions.

***Rietveld Refinement & Indexing Workshops:**

Basic: 28–30 September 2015 / Advanced: 1–2 October 2015

Powder Pattern Indexing and Rietveld structural refinement techniques are complementary and are often used to completely describe the structure of a material. Successful indexing of a powder pattern is considered strong evidence for phase purity. Indexing is considered a prelude to determining the crystal structure, and permits phase identification by lattice matching techniques. This workshop introduces the theory and formalisms of various indexing methods and structural refinement techniques. One unique aspect of this workshop is the extensive use of computer laboratory problem solving and exercises that teach method development in a hands-on environment.

Take the three-day basic workshop, the two-day advanced workshop or both together for a full week of hands-on training. The ICDD basic Rietveld workshop is a pre-requisite for attending the advanced workshop. A basic understanding of crystallography is also required.



Don't miss the opportunity to meet with our faculty, offering knowledge in a wide range of industries and applications. You'll meet seasoned professionals with experience in metals, microelectronics, thin films, indexing, polymers, organic chemistry and much more. Featuring live instruments for the XRF & XRD Clinics!

Register Today at WWW.ICDD.COM/EDUCATION



* See the ICDD web site for prerequisites for advanced courses.

FOR MORE INFORMATION CONTACT

Eileen Jennings, Education Coordinator

Tel: 610.325.9814 Fax: 610.325.9823

Email: clinics@icdd.com

LOCATION

ICDD Headquarters, 12 Campus Boulevard
Newtown Square, Pennsylvania 19073-3273 U.S.A.



EDITORIAL

- Vanessa K. Peterson and Nathan A.S. Webster Proceedings of the 2014 Australian X-ray Analytical Association Workshops, Conference, and Exhibition S1

TECHNICAL ARTICLES

- Pamela S. Whitfield Diffraction studies from minerals to organics: lessons learned from materials analyses S2
- Damian B. Gore, Mark P. Taylor, R. Gary Pritchard and Kirstie A. Fryirs On-site teaching with XRF and XRD: training the next generation of analytical X-ray professionals S8
- Thomas A. Whittle and Siegbert Schmid Structural investigation of tungsten bronze-type relaxor ferroelectrics in the $Ba_xSr_{3-x}TiNb_4O_{15}$ system S15
- Nobuo Ishizawa Calcite V: a hundred-year-old mystery has been solved S19
- A.M. Paradowska, N. Larkin, H. Li, Z. Pan, C. Shen and M. Law Neutron diffraction residual stress measurements of welds made with pulsed tandem gas metal arc welding (PT-GMAW) S24
- Sarah J. Kelloway, Colin R. Ward, Christopher E. Marjo, Irene E. Wainwright and David R. Cohen Calibration for ED-XRF profiling of coal cores for the Itrax Core Scanner S28
- Moshiul Alam, Tracey Hanley, Wei Kong Pang, Vanessa K. Peterson and Neeraj Sharma Comparison of the so-called CGR and NCR cathodes in commercial lithium-ion batteries using *in situ* neutron powder diffraction S35
- Damian B. Gore and Ian Snape 50 kGy of gamma irradiation does not affect the leachability of mineral soils and sediments S40
- Marco Sommariva, Milen Gateshki, Jan-André Gertenbach, Joerg Bolze, Uwe König, Bogdan Ștefan Vasile and Vasile-Adrian Surdu Characterizing nanoparticles with a laboratory diffractometer: from small-angle to total X-ray scattering S47
- Nathan A. S. Webster, Mark I. Pownceby, Ian C. Madsen, Andrew J. Studer and Justin A. Kimpton *In situ* diffraction studies of iron ore sinter bonding phase formation: QPA considerations and pushing the limits of laboratory data collection S54
- Wei Kong Pang, Vanessa K. Peterson, Neeraj Sharma, Je-Jang Shiu and She-huang Wu Structure of the $Li_4Ti_5O_{12}$ anode during charge-discharge cycling S59
- Markus Gräfe, Craig Klauber, Bee Gan and Ryan V. Tappero Synchrotron X-ray microdiffraction (μ XRD) in minerals and environmental research S64
- Nathan A. S. Webster, Chris D. Ling and Frank J. Lincoln Structure–property relationships in fluorite-type Bi_2O_3 – Yb_2O_3 – PbO solid-electrolyte materials S73
- Uwe König, Thomas Degen and Nicholas Norberg PLSR as a new XRD method for downstream processing of ores: – case study: Fe^{2+} determination in iron ore sinter S78

Anita M. D'Angelo, Nathan A. S. Webster and Alan L. Chaffee	Characterisation of the phase-transformation behaviour of $\text{Ce}_2\text{O}(\text{CO}_3)_2 \cdot \text{H}_2\text{O}$ clusters synthesised from $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and urea	S84
Jian Li, Robbie G. McDonald, Anna H. Kaksonen, Christina Morris, Suzy Rea, Kayley M. Usher, Jason Wylie, Felipe Hilario and Chris A. du Plessis	Applications of Rietveld-based QXRD analysis in mineral processing	S89
Stephen H. Ogilvie, Samuel G. Duyker, Peter D. Southon, Vanessa K. Peterson and Cameron J. Kepert	Host-guest adsorption behavior of deuterated methane and molecular oxygen in a porous rare-earth metal-organic framework	S96
Joel N. O'Dwyer, James R. Tickner and Greg J. Roach	Predicting the accuracy of mineral phase analysis by X-ray diffraction using Monte Carlo modelling	S102

Powder Diffraction

An International Journal of Materials Characterization

Editor-in-Chief

Camden Hubbard
Applied Diffraction Services
110 Crestview Lane
Oak Ridge, Tennessee 37830, U.S.A.
camden.hubbard@me.com

Managing Editor

Nicole M. Ernst Boris
International Centre for Diffraction Data
12 Campus Boulevard
Newtown Square, Pennsylvania 19073-3273, U.S.A.
boris@icdd.com

Editor for New Diffraction Data

Soorya Kabekkodu
International Centre for Diffraction Data
12 Campus Boulevard
Newtown Square, Pennsylvania 19073-3273, U.S.A.
kabekkodu@icdd.com

Editors

Xiaolong Chen
Institute of Physics
Chinese Academy of Sciences
No. 8 Nansanjie, Zhongguancun, Haidian District,
Beijing 100190,
China
xlchen@iphy.ac.cn

José Miguel Delgado
Universidad de Los Andes
Facultad de Ciencias
Departamento de Química
Lab. de Cristalografía
Mérida 5101
Venezuela
miguel@ula.ve

Norberto Masciocchi
Università dell'Insubria
Dipartimento di Scienza e Alta Tecnologia
via Valleggio 11
Como 22100
Italy
norberto.masciocchi@uninsubria.it

Editors for Crystallography Education

James Kaduk
Poly Crystallography Inc.
423 East Chicago Avenue
Naperville, Illinois 60540-5407, U.S.A.
Kaduk@polycrystallography.com

Brian H. Toby
Argonne National Laboratory
Advanced Photon Source
9700 S. Cass Ave., Bldg. 401/B4192,
Argonne, Illinois 60439-4856, U.S.A.
brian.toby@anl.gov

International Reports Editor

Winnie Wong-Ng
Materials Measurement Science Division
National Institute of Standards and Technology
100 Bureau Drive, Mail Stop 8520
Gaithersburg, MD 20899-8520, U.S.A.
winnie.wong-ng@nist.gov

Calendar of Meetings and Workshops Editor

Gang Wang
Institute of Physics
Chinese Academy of Sciences
No. 8 Nansanjie, Zhongguancun, Haidian District,
Beijing 100190,
China
gangwang@iphy.ac.cn

On the Cover: Featured AXAA Proceedings images From Minerals to Materials (Courtesy of Markus Gräfe, Craig Klauber, Bee Gan, Ryan V. Tappero, Moshui Alam, Tracey Hanley, Wei Kong Pang, Vanessa K. Peterson, Neeraj Sharma, Anita D'Angelo, Nathan A.S. Webster, Alan L. Chaffee, Stephen H. Ogilvie, Samuel G. Duyker, Peter D. Southon, and Cameron J. Kepert).

Powder Diffraction is a quarterly journal published by the JCPDS-International Centre for Diffraction Data through Cambridge University Press.

Powder Diffraction is a journal of practical technique, publishing articles relating to the widest range of application—from materials analysis to epitaxial growth of thin films and to the latest advances in software. Although practice will be emphasized, theory will not be neglected, especially as its discussion will relate to better understanding of technique.

Submit manuscripts online at <http://mc.manuscriptcentral.com/pdj>. See the instructions on submitting your manuscript linked on that page. The editors will consider all manuscripts received, but assume no responsibility regarding them. There is no publication charge.

Most proofs are handled via email at kdaly@cambridge.org. Proofs and all correspondence concerning papers in the process of publication can also be addressed to: Production Editor, *Powder Diffraction*, Cambridge University Press, 32 Avenue of the Americas, New York, NY 10013-2473, U.S.A. Please include the job number in all correspondence.

For advertising rates and schedules contact Cambridge University Press Advertising Sales. Orders, advertising copy, and offset negatives should be sent to: Advertising Sales, Cambridge University Press, 32 Avenue of the Americas, New York, NY, 10013-2473, U.S.A.; Phone: 212-924-3900; Fax: 212-924-3900. Email: USAdSales@cambridge.org.

Subscription Prices 2014

	Print & Online	Online
Individual (U.S. & Canada)	\$150	\$121
Individual (outside U.S. & Canada)	\$160	\$116
Student	N/A	\$30
Institutional or Library	\$273	\$181

Subscription rates to Eastern Hemisphere include air freight service.

Back-Number Prices. 2014 single copies: \$75.

Subscription, renewals, and address changes should be addressed to Subscription Fulfillment, *Powder Diffraction*, Cambridge University Press, 100 Brook Hill Drive, West Nyack, NY 10013-2113 (for U.S.A., Canada, and Mexico); or Cambridge University Press, The Edinburgh Building, Shaftsbury Road, Cambridge, CB2 8RU, Cambridge, England (for UK and elsewhere). Allow at least six weeks advance notice. For address changes please send both old and new addresses and, if possible, include a mailing label from the wrapper of a recent issue.

Claims, Single Copy Replacement, Back Volumes, and Reprints: Missing issue requests will be honored only if received within six months of publication date (nine months for Australia and Asia). Single copies of a journal may be ordered and back volumes are available in print or microform. Individual subscribers please contact Subscription Fulfillment, *Powder Diffraction*, Cambridge University Press, 100 Brook Hill Drive, West Nyack, NY 10013-2113. Phone: 845-353-7500; Toll free: 800-872-7423; Fax: 845-353-4141. Email: subscriptions_newyork@cambridge.org.

Powder Diffraction (ISSN: 0885-7156) is published quarterly (4X annually) by the JCPDS-International Centre for Diffraction Data through Cambridge University Press, 32 Avenue of the Americas, New York, NY 10013-2473. POSTMASTER: Send address changes to *Powder Diffraction*, Cambridge University Press, 100 Brook Hill Drive West Nyack, NY 10994-2113, USA. Periodicals postage paid in New York, NY and additional mailing offices.

Permission for Other Use: Permission is granted to quote from the journal with the customary acknowledgment of the source. To reprint a figure, table, or other excerpt requires the consent of one of the authors and notification to Cambridge University Press.

Requests for Permission: No part of this publication may be reproduced in any forms or by any means, electronic, photocopying, or otherwise, without permission in writing from Cambridge University Press. Policies, request forms, and contacts are available at: <http://journals.cambridge.org/action/rightsAndPermissions>. Permission to copy (for users in the U.S.A.) is available from Copyright Clearance Center: <http://www.copyright.com>. Email: info@copyright.com.

Document Delivery and Online Availability: Abstracts of journal articles published by Cambridge University Press are available from Cambridge Journals Online (<http://journals.cambridge.org/action/displayJournal?jid=PDJ>).

Copyright © 2014 JCPDS- International Centre for Diffraction Data, 12 Campus Blvd., Newtown Square, PA 19073-3273, U.S.A. All rights reserved. www.icdd.com/products/journals.htm



GRANT-IN-AID Program



JOIN ICDD'S ELITE GROUP OF SCIENTISTS

who contribute experimental powder diffraction patterns to the Powder Diffraction File

AS A MEMBER OF THIS ELITE GROUP, YOU WILL RECEIVE THE FOLLOWING BENEFITS:

- Financial support to aid current research
- Publication of pattern(s), if accepted by the Editorial Department, in the Powder Diffraction File
- Receive calibration standards
- Purchase certain products at reduced prices
- Web-based access to the list of compounds in the ICDD master database—includes published patterns, as well as patterns still in the editorial process
- First-time grantees receive a complimentary one-year subscription to *Powder Diffraction*

DISTINGUISHED GRANTEES



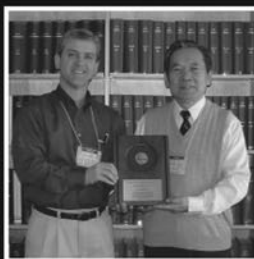
2013: (L)
Professor Xinkan Yao



2010: (R)
Dr. Tom Blanton
presenting to
Professor Bogdan Lazoryak



2007: (L)
Dr. Miguel Delgado
presenting to
Dr. Sergei Kirik



2004: (R)
Dr. Tom Blanton
presenting to
Professor Shao-Fan Lin



2001: (L)
Dr. Tom Blanton
presenting to
Professor Evgeny Antipov



1998: (R)
Dr. Ekkehart Tillmanns

For over 50 years, ICDD has supported a well-developed program of grants to researchers around the world.

One of our main objectives is to expand the range of reference materials by producing and cataloging high-quality X-ray diffraction patterns in our internationally renowned database, the Powder Diffraction File. Thanks to the longevity of this program, these contributions account for approximately a quarter of the current experimental file. ICDD awards financial support to qualified investigators in the form of grants-in-aid on a competitive proposal basis. The duration of a grant is 12 months with two cycles per year. Cycle I begins 1 April and Cycle II begins 1 October.

Geographic Locations of Grants for the Past 15 Years:

Argentina	Malaysia
Austria	Netherlands
Canada	P.R. of China
Chile	Poland
Columbia	Portugal
Czech Republic	Russia
Estonia	Spain
Finland	Switzerland
France	Taiwan
Germany	Tunisia
India	Ukraine
Israel	United Kingdom
Italy	United States
Japan	Uruguay

Total Proposals Funded for the Past 15 Years: **720**

Could you be the next Distinguished Grantee?
www.icdd.com/grants

For more information on ICDD's Grant-in-Aid, visit www.icdd.com/grants