NIST Special Publication 846

Accuracy in Powder Diffraction II

Proceedings of the International Conference May 26–29, 1992

E. Prince and J. K. Stalick, Editors

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[1]At Boulder, CO 80303.
[2]Some elements at Boulder, CO 80303.

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Foreword

Few areas of research endeavour in the physical sciences have experienced a renaissance on such a scale, and few have had such an impact on related disciplines during the past 15 years, as has powder diffraction. Sensing this imminent revolution back in 1979, the U. S. National Bureau of Standards (now NIST) hosted a conference entitled Accuracy in Powder Diffraction. The proceedings of this symposium were subsequently published as NBS Special Publication 567 and this document is still widely quoted in the literature, more than 10 years later!

In the years since this benchmark meeting, new generations of high resolution and intense sources of X-rays and neutrons have been designed and built, new and exciting methods for data collection and analysis have been developed, and powerful computers have come within reach of, if not on, scientists' desks. Never before have so many previously neglected materials come under such close scrutiny, and never before has it been possible to study these materials under such a wide range of non-ambient conditions. Diffraction patterns previously collected only for phase identification or unit cell determination are now used to study such things as the subtleties of electron density distribution, the mechanisms of phase transformations, and the distribution of cation ionization states.

In the light of this renaissance, the IUCr Commission on Powder Diffraction (CPD), in conjunction with the National Institute of Standards and Technology, JCPDS–International Centre for Diffraction Data, and the International Union of Crystallography, organized a second conference, Accuracy in Powder Diffraction II, as a continuation of the series started in 1979. Six sessions provided the framework for APD-II: phase identification and quantification; accuracy and standards; new developments in software and data analysis; profile fitting, decomposition, and microstructural effects; novel applications and structural science; new developments in hardware, including detectors, and studies under non-ambient and time-resolved conditions.

The scientific sessions incorporated keynote, invited, oral and poster papers, a tribute to the late W. Parrish, and two workshops organized by the JCPDS–ICDD, entitled *ICDD round robin on powder diffractometer sensitivity* and *Indexing methods*. In addition, the program for the last session of the meeting was specifically designed to interface smoothly with the Workshop on X-ray and Neutron Diffraction at High Pressure, organized by the High pressure Group of the IUCr, which was held in Washington, DC, at the Geophysical Laboratory of the Carnegie Institution of Washington. Many attendees obtained substantial benefit from attending both of these meetings.

Generous support for the attendance of young scientists and invited speakers was obtained from the IUCr and the JCPDS–ICDD, respectively. It need hardly be added that the continued financial support of both of these organizations is extremely valuable to, and very much appreciated by the powder diffraction community in general and the APD-II Organizing Committee in particular. The cooperation and generosity of the NIST in providing support in the form of both personnel and facilities was essential for the organization of the meeting, and for this we are also very grateful.

I take the opportunity here to express my thanks to the members of the Scientific Program Committee for their hard work and cooperation; their efforts resulted in an enlightening and fruitful meeting.

R.J. Hill
Chairman, Scientific Program Committee

Acknowledgments

The timely publication of this volume of proceedings was made possible by the cooperation of the authors, who provided machine-readable text, many of them in LaTeX or Wordperfect, so that the labor involved in producing camera-ready copy was minimal. We are grateful to Carol O'Connor for invaluable assistance in preparing the text for the printer. The success of the conference was assured by the efforts of the international program committee, ably chaired by Rod Hill, and for the smooth running of the conference we are indebted to Kathleen Kilmer and her staff in the NIST conference office, particularly Tammie Grice and Lori Phillips. The conference would not have occurred at all without the impetus of Ray Young and the IUCr Commission on Powder Diffraction and the generous sponsorship of the International Union of Crystallography and the JCPDS–International Centre for Diffraction Data.

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Abstract

The proceedings of the international conference Accuracy in Powder Diffraction II present the invited papers and abstracts of the papers contributed to the conference, which was held at NIST, Gaithersburg, Maryland, during May 26-29, 1992. The conference was organized by the Commission on Powder Diffraction of the International Union of Crystallography, and was jointly sponsored by NIST, JCPDS - International Centre for Diffraction Data and the International Union of Crystallography. The proceedings contain 25 invited papers and 73 contributed abstracts. The program of the conference was divided into six topics: Phase Identification and Quantification; Accuracy and Standards; New Developments in Software and Data Analysis; Profile Fitting, Decomposition and Microstructural Effects; Novel Applications and Structrual Science; and New Developments in Hardware, Including Detectors, and Studies under Non-ambient and Time-resolved Conditions. A ceremonial session was devoted to a tribute to the late William Parrish and his contributions to powder diffraction. In addition, there were two tutorial workshops organized by the JCPDS - International Centre for Diffraction Data, one on diffractometer sensitivity and one on automatic indexing methods.

O2.1 ACCURATE DETERMINATION OF UNIT-CELL PARAMETERS USING AN INTERNAL STANDARD REFERENCE MATERIAL AND WHOLE-PATTERN FITTING. Hideo Toraya, Ceramics Research Laboratory, Nagoya Institute of Technology, Asahigaoka, Tajimi 507 Japan

A new technique for peak-shift correction achieved the accuracy of a few p.p.m. in routinely determining the unit-cell parameters of a sample with internal standard reference material (Toraya & Kitamura, J. Appl. Cryst., 23, 282-285, 1990; Toraya & Parrish, PICXAM, 1991). This algorithm is based on 1) the simultaneous least-squares determination of both unit-cell parameters and unknown parameters in the calibration function and 2) the use of observed profiles with symmetric shape. The first idea in the above two has already been realized by the whole-powder-pattern fitting without reference to a structure model (Toraya, Advances in Ceramics, Vol 21, 811-819, 1987). However, the profile asymmetry usually observed in conventional powder diffractometry induced a strong parameter correlation between the peak positions (unit-cell parameters) and parameters in the function for modeling profile asymmetry, and the required accuracy could not be realized. Recent use of a high-resolution powder diffractometer and a synchrotron radiation source make it possible to obtain nearly symmetric profile and greatly improve the accuracy of determining the peak positions. The use of whole-powder-pattern fitting has a great advantage in that the method can easily be extended to the analyses of samples with low crystallographic symmetry. In the present study, the unit-cell parameters of samples of cubic to orthorhombic symmetry were measured by using whole-powder-pattern fitting on the above two bases. The procedure will have potential uses in the routine detrmination of unit-cell parameters with both conventional tube-generated X-rays and synchrotron radiation.

P2.2 INSTRUMENT ALIGNMENT METHODS FOR RESIDUAL STRESS MEASUREMENTS BY MEANS OF NEUTRON DIFFRACTION. P.C. Brand and H.J. Prask, National Institute of Standards and Technology, Gaithersburg, MD 20899, U.S.A.

Neutron powder diffraction in general, and residual stress measurements by means of neutron diffraction in particular, require very well aligned instrumentation.

Unlike regular powder diffraction where the specimen is fully immersed in the neutron beam, stress measurements by means of neutron diffraction require the presence of small beam apertures that define the size of the gauge volume in the specimen. It is the gauge volume for which one determines the stress state during a stress measurement.

The correct positioning of the beam apertures is crucial for successfully applying the stress measurement technique. Therefore we have developed a sequence of alignment procedures to assure correct positioning. These employ diffraction in a small test specimen situated at a known location on the neutron diffractometer, and a new analysis method of the diffraction data taken from the test specimen. These will be presented. The presented alignment methods have proved extremely helpful in obtaining very well aligned beam apertures in a reproducible manner.

P2.1 A UNIVERSAL FILE FORMAT FOR POWDER DIFFRACTION DATA ARCHIVE AND INTERCHANGE: THE IUCr CRYSTALLOGRAPHIC INFORMATION FILE (CIF) FORMAT. B. H. Toby, Air Products & Chemicals Inc, 7201 Hamilton Boulevard, Allentown, PA 18195, USA; J. I. Langford, University of Birmingham, School of Physics & Space Research, Birmingham B15 2TT, UK; and S. R. Hall, Crystallography Centre, University of Western Australia, Nedlands 6009, Australia.

Powder diffraction data items have been defined for use with the Crystallographic Information File (CIF) approach to data storage and exchange. These will permit the ready exchange of processed and raw powder data generated by diffractometers of every type: commercial sealed-tube instruments; CW synchrotron; neutron; energy-dispersive; and time-of-flight. International collaboration is vital to ensure that the definitions meet the needs of the powder diffraction community.

The Crystallographic Information File (CIF) has been developed by the IUCr for storage of crystallographic data ranging from measured intensities through structural diagrams and manuscript text [S. R. Hall, F. H. Allen & I. D. Brown, *Acta Cryst. A47*, 655-685.] A CIF is composed of ASCII characters which may edited with a normal text editor, and transmitted via electronic networks (e.g. email or ftp). A CIF is completely self-descriptive in that data items are identified by unique tags (referred to as a data name). These data names may be defined locally or globally. Global data items (i.e. those used outside the local environment) are carefully defined in a CIF data name dictionary that is also electronically stored in a CIF-like format.

Powder diffraction data are usually measured by computer-controlled diffractometer or densitometer and are stored in a data file format specific to the measurement instrument. Typically these data are then processed using computer programs modified to use data in the local format. If the data will be published or archived, the diffractogram will usually be reduced to a peak table of position and intensity values. Apart from the need to standardise the data processing software, the need for the global exchange of unprocessed powder diffraction data is increasing. This need arises because of the use of whole pattern methods for (i) crystalline phase identification and quantification, (ii) ab initio structure determination, (iii) structural refinements, and (iv) characterization of materials with limited ordering (e.g. polymers and clays), where the diffractogram cannot be represented as a set of peaks.

Definitions for CIF data items used in powder diffraction are proposed. These are in addition to those data items in the proposed 1991 Core CIF Dictionary. Discussion of these definitions is to be encouraged prior to their formal adoption at the 1993 IUCr Congress.

P2.3 A REFERENCE SPECIMEN FOR HIGH RESOLUTION LINE PROFILE ANALYSIS. PREPARATION, CHARACTERIZATION AND APPLICATION. J.G.M. van Berkum, G.J.M. Sprong, Th.H. de Keijser, E.J. Sonneveld, A.C. Vermeulen & R. Delhez. Delft University of Technology, Laboratory of Materials Science, Rotterdamseweg 137, 2628 AL Delft, The Netherlands.

High resolution line profile analysis requires a high quality standard reference specimen to characterize the instrumental line profile. A universal reference has to meet very stringent criteria, of which negligible structural broadening is the most important. Si SRM 640a/b powder has been selected as an appropriate starting material. Reference specimens have been prepared according to a standardized procedure, which includes (i) removal of the smallest particles ($<5 \mu m$), (ii) sedimentation in 2-propanol of a mono-layer of particles onto a Si (510) single crystal substrate (for low background) and (iii) annealing for 20 h at 1273 K. The sedimented powder sticks to the substrate and is very homogeneously distributed. Loose fractionated powder, annealed in a rotating ampulla, can be applied as an internal standard.

Using a conventional powder diffractometer the reference specimens yield narrower line profiles than those reported in the literature (down to $0.050° 2\theta$ full width at half maximum) and the breadth vs. 2θ plots show very smooth curves. As a comparison LaB_6 SRM 660, also sedimented onto a Si substrate (but no other treatment), yields systematically broader lines over the complete 2θ range and the breadth vs. 2θ plot shows deviations from a smooth curve due to {hkl}-dependent structural broadening. Crystal statistics is the limiting factor for the line profile reproducibility. For a spinning specimen with $2 - 4 \times 10^4$ particles per mm^2 the integral intensity and the breadth can be reproduced within 1%. An improvement is possible by preparing thicker specimens, having several layers of powder particles, but only at the cost of extra 'transparency' broadening. Essentially mono-layered non-spinning specimens are not suited as a reference.

Using the new reference specimens line profiles with a very small amount of size and strain broadening can be interpreted reliably. As an example, line profiles from ball-milled particles (1-10 μm) of various materials have been studied. Although the integral breadths of the measured lines may be smaller than $0.1° 2\theta$ at low angles, they can be analysed reliably using Stokes deconvolution. Besides high resolution line profile analysis, the new class of standard specimens also provides a means for assessing the state of any powder diffractometer. The smallest deviations from the perfect alignment, from the ideal shape of a focus or from the average room or cooling-water temperature result in detectable breadth changes.

P2.4   SELFCORRECTING METHOD FOR PRECISE DETERMINATION OF LATTICE PARAMETERS. M. Ya Gamarnik, Institute of Geochemistry and Physics of Minerals, Academy of Sciences, Palladin Pr. 34, Kiev 252680, Ukraine

The standardless method is proposed for lattice parameters determination by X-ray diffractometer. Goniometer aberrations, distorting Bragg's reflections, can be accounted for by the method. The displacement of the sample reflecting plane from the goniometer axis, the zero detector position, the longitudinal and transversal components of goniometer eccentricity are determined from reflections locations for both large (analytical reflections) and small (correcting reflections) Bragg angles. These aberrations are calculated from the equation systems including also the lattice parameters as unknown quantities. The method accuracy is determined by that of the angle measurement and by the positions of analytical and correcting reflections. The method can be used for polycrystal and monocrystal samples of any crystallographic system.

P2.6   ACCURACY IN POWDER DIFFRACTION: OPTIMISATION OF DATA COLLECTION STRATEGIES. W.I.F.David, ISIS Facility, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon., OX11 0QX, U.K.

X-ray and neutron constant wavelength powder diffraction are conventionally collected in step scan mode with constant time increments or with a constant monitor count. From an instrumental viewpoint these methods are clearly the simplest - additionally, of course, the resulting pattern has a Bragg peak intensity variation that is similar to that obtained by photographic methods. For neutron diffraction data this generally is an adequate procedure since the high angle Bragg peaks do not suffer from a form-factor fall-off. However, in X-ray powder diffraction the atomic scattering factor variation as a function of $\sin\theta/\lambda$ may lead to intensity differences that exceed two orders of magnitude between low and high angle data. Standard least-squares for X-ray profile refinement is often dominated by fitting the first few Bragg peaks (which contain the least structural information!). The principle of optimised powder diffraction data collection strategy that will be discussed is similar to well-known leverage techniques that are used in single crystal data collection. Simple theoretical models will be presented and initial results discussed.

P2.5   USE OF AN AUTOMATED POWDER DIFFRACTOMETER TO PERFORM LATTICE PARAMETER REFINEMENT. R. A. Newman, Analytical Sciences Laboratory, 1897 G Building, Dow Chemical, Midland, Michigan 48667, U. S. A.

It is well known that carefully performed experiments using specialized powder diffraction instrumentation to analyze pure, crystalline materials can yield lattice parameters with a precision of $1x10^{-5}$ or better. However, this is seldom the case when using standard laboratory diffraction equipment, especially Bragg-Brentano geometry powder diffractometers. The errors associated with the reflection geometry, sample size/preparation, etc. for typical diffractometers limit lattice refinement performed on these units to precision on the order of $1x10^{-4}$ in the best cases.

At Dow, we have improved our ability to perform diffractometer-based lattice refinements by using the combination of [1] a Siemens D-500 diffractometer equipped with an incident beam monochromator, [2] the use of zero-scatter sample holders, and [3] our in-house developed profile-fitting software. With this system, we are able to obtain data from a $K\alpha_1$ source, which greatly simplifies the diffraction pattern. The use of a shallow depth zero-scatter sample holder reduces the effect of sample transparency and flat sample errors. Finally, the ability to perform interactive profile decomposition base on a split Pearson VII algorithm improves our accuracy in peak centroid determination. Utilizing this approach, we are able to routinely obtain lattice refinement results on the order of $1x10^{-4}$. The various steps in the data collection and analysis procedure will be illustrated, and examples of lattice refinements will be shown.

P2.7   ON THE PRECISION AND ACCURACY OF POWDER DIFFRACTION STUDIES OF KAOLINITE AND DICKITE. Ľ. Smrčok, Institute of Inorganic Chemistry, Slovak Academy of Sciences, 842 36 Bratislava, Czechoslovakia.

Kaolinites are known to exist in four polytypic forms which differ only in the stacking mode of simple layers formed by SiO_4 tetrahedral and $Al(O,OH)_6$ octahedral sheets. Since kaolinite, in contrast to dickite, has not, with one exception, yielded single crystals suitable for structure analysis, (the results are, however, of lower quality) all structure refinements carried out so far have been based on powder data. Such refinements are hampered mainly by heavy overlap of the majority of individual reflections (kaolinite is triclinic) and by kaolinite's extreme tendency to orient preferentially. In order to evaluate the precision and/or accuracy of the refined structure parameters, we compared the interatomic distances $d(Si-O)$ and $d(Al-O)$ calculated from the data reported in ten powder refinements of both well ordered and partially disordered kaolinite specimens. The main tool used in our considerations were median-based box-and-whisker plots. We have found, that the $d(Si-O)$ are within the interval $< 1.43, 1.87 >$Å while $d(Al-O)$ vary from $< 1.64, 2.12 >$Å; the expected values being 1.617 and 1.91 Å, respectively. A comparison of these individual refinements with two reliable single crystal analyses of dickite has shown that the spread of distances is in only one case comparable with the single crystal results. Several authors have reported rather precise but inaccurate distances, or vice versa. As far as the medians are concerned, all refinements have provided very close results, but the sizes of the boxes vary significantly from one refinement to another. Potential sources of difficulties are (not ordered according to their relative importance): preferred orientation wrongly accounted for; weights emphasizing the role of small intensities, which dominate in the region of heavy overlap and are measured frequently with very limited precision; small ratio of the number of Bragg reflection to the number of refined structure parameters; and limited ability of the method to decompose unambiguously profiles that are heavily overlapped. It is rather surprising that the accuracy of the interatomic distances is almost independent of the presence or absence of disorder in the structure.

O3.1 THE FAST ITERATIVE PATTERSON SQUARING (FIPS) METHOD - AN IMPROVED TREATMENT OF SEVERELY OVERLAPPING REFLECTIONS IN A POWDER PATTERN FOR THE APPLICATION OF DIRECT METHODS. M. Estermann and V. Gramlich, Institute of Crystallography, ETH, CH-8092 Zürich. Switzerland.

Single crystal techniques have been used to solve a number of structures *ab initio* from powder data (see, for example, refs. (1) and (2), and references therein).

Obviously, these techniques require a single-crystal-like data set, which has to be extracted from the powder pattern. Extraction programs cope well with partial overlap of diffraction intensity in the powder pattern, but not with severe or exact overlap. Usually the intensity ratio of these severely overlapping reflections is set to one (equipartitioning).

Unfortunately, direct methods, which are so successful for routine single crystal structure determination, often fail when used with an extracted powder data set. The limiting factors are the complexity of the crystal structure and the number of severely or exactly overlapping reflections in the powder pattern.

In our laboratory, we have recently developed a new method, the *Fast Iterative Patterson Squaring* (FIPS) method (3,4), for unravelling the intensities of severely or exactly overlapping reflections. Starting from an equipartitioned data set, a Patterson map is generated, each point in the map is squared, this new map is backtransformed to obtain new Fourier coefficients, and these coefficients are then extrapolated to give a new set of $|F(hkl)|^2$ and a new intensity distribution for the overlapping reflections (non-overlapping ones remain unchanged). The cycle is repeated until the intensity statistics of the overlapping reflections approximate those of the non-overlapping ones. Tests with powder data obtained from materials with known crystal structures proved to be successful.

The molecular sieve SAPO-40 of unknown structure, which could only be synthesized in the form of a polycrystalline powder, provided a real test case for the FIPS method (5). Approximately 65% of all the reflections were affected by severe overlap, and the structure could not be solved from the equipartitioned data. However, after the intensities had been redistributed using the FIPS method, the structure was solved by direct methods.

(1) McCusker, L. (1988). *J. Appl. Cryst.* 21, 305-310.
(2) Hiraguchi *et al.* (1991). *J. Appl. Cryst.* 24, 286-292.
(3) Estermann, M. (1991). Thesis.
(4) Estermann, M. and Gramlich, V. (1992). in preparation.
(5) Estermann, M. and McCusker, L. (1992). in preparation.

O3.2 COMBINED RIETVELD AND SHORT RANGE ORDER PARAMETER REFINEMENT. J. Schneider, Institut für Kristallographie und Mineralogie, Universität München, Theresienstrasse 41, D-8000 München 2 Germany.

Although the Rietveld method[1] has become a widely accepted technique in powder diffraction to refine structural parameters from Bragg peak profiles, less attention has been given to refinement of concurrent diffuse disorder gcattering. Substitutional disorder in binary alloys produces a modulation of monotonic Laue scattering intensity if short range order is present. For cubic polycrystalline alloys this may be written as a series expansion[2]:

$$I_{SRO} = N \ x_A x_B \ (f_A - f_B)^2 \ \sum_i c_i \alpha_i \sin(kr_i)/(kr_i) \qquad (1)$$

N is the number of lattice sites, x_A and x_B are the fractions of the A and B components, respectively, f_A and f_B their formfactors (scattering lengths), c_i is the number of neighbors in shell i, r_i it's radius, $k = 4\pi\sin\Theta/\lambda$ and α_i are the well known Warren-Cowley short range order parameters. Eq. (1) (i=1..6) was incorporated into the local PC version[3] of a standard Rietveld program[4].

Application of the program to neutron data[5] of Na deficient Na_xTl_{1-x}, x = 0.47 showed that the observed diffuse intensity can well be described by eq. (1). The major contribution arises from a term which yields $r_1 = 3.227(9)$ Å. This agrees well with the next neighbor distance $d = \sqrt{3} \ a_0 = 3.2250(4)$ Å derived from the Rietveld refinement. Assuming that the heavy Tl atoms do not take part in substitutional disorder, this result suggests short range order between Na atoms (and vacancies) on the Na sublattice.

[1] Rietveld, H. M., J. Appl. Cryst. 2 (1969) 65.
[2] Warren, B. E., X-ray diffraction, Addison-Wesley 1969.
[3] Schneider J., Dinnebier R. E., Material Science Forum 79/82 (1991) 277.
[4] Sakthivel A., Young R. A., Int. Workshop on the Rietveld method, Petten 1989.
[5] Schneider J., Material Science Forum 27/28 (1988) 63.

P3.1 USE OF THE CSD PROGRAM PACKAGE FOR STRUCTURE DETERMINATION FROM POWDER DATA. L. G. Akselrud, P. Yu. Zavalii, Yu. N. Grin, V. K. Pecharski. Institute of Inorganic Chemistry, Lviv State University, 290005 Lviv, Ukraine & B. Baumgartner, E. Wölfel, STOE & Cie, 6100 Darmstadt, Germany

The CSD program package has been developed to combine the possibilities of single crystal and powder diffractometry for crystal structure research and offers to the user tools for solution, refinement and interpretation of the crystal structure independent of the data collection method. It runs on a PC XT/AT/PS2 or fully compatible computer systems under DOS3.3 and higher.

The power of this program system has been tested using the powder data of various samples with already known structures. These crystal structures have been investigated using STOE powder diffractometers for data collection and CSD as the computational system, starting from reflection indexing and followed by structure solution and integrated-intensity or full-profile structure refinement. Different kinds of data collection (transmission and Bragg-Brentano geometries) have been compared in respect to their advantages for structure solution. The samples comprise both inorganic and organic compounds with crystal systems ranging from triclinic to cubic, thus presenting usual experimental situations. These include fully overlapped peaks as a result of symmetry of the unit cell, partially overlapped peaks dependent on unit cell parameter relations, and different variation of peak-half-widths with theta for different reflection zones.

The precision and accuracy of these results are discussed.

P3.2 SOFTWARE PACKAGE PATCOM FOR SIMULATION AND MANIPULATION OF POWDER DIFFRACTION PATTERNS AND STANDARDS. Ya. E. Cherner, Institute of Physics, 194 Stachki Ave., Rostov-on-Don, 344104, Russia.

The PATCOM software package is intended for simulating, comparing, displaying, and printing powder diffraction patterns of single- or multiphase samples and solid solutions using crystal structure data for the substance or its isomorph. It consists of five programs.

SCREEN is a program for preparation of the input files. The SITES and HKL programs calculate, by means of symmetry operations, the atomic position multiplicities and coordinates of all atomic sites, as well as the Miller indices, d-spacings, angular positions, and multiplicity factors of all lines within the desired angle range.

INTENS computes both the integrated and relative intensities of Bragg reflections. It also calculates the unit cell weight, volume, density, and the mass absorption factor. INTENS allows the creation of lattice vacancies, and can place up to 5 different atoms (ions) into a single atomic position.

PICTURE is an interactive color graphics program for:
- displaying or printing up to 5 theoretical or measured patterns, or powder diffraction standards simultaneously as a 2-, or 3-dimensional picture (line- and continuous diagrams are available);
- simulating and storing the patterns of multiphase substances;
- comparing patterns of different origin by means of superposition and/or subtraction.

Five profile functions are available for peak shape simulation and the profile parameters may be changed. STRART enables crystal structure to be displayed or plotted.

Options include rotation of a structure model around any direction, shift of any atoms and recalculation of the theoretical pattern for a modified atomic arrangement, drawing of the unit cell projection on a desired plane, etc.

P3.3 SOFTWARE PACKAGE FOR POWDER DIFFRACTION DATA PROCESSING. Ya.E. Cherner, Institute of Physics, 194 Stachki Ave., Rostov-on-Don, 344104, Russia.

The software package PD-ASSISTANT is intended for handling measured powder diffraction patterns. It consists of four programs.

The PATTR program offers many facilities for displaying and treating patterns. It also creates the input parameters files for the BCGR and PEFIT programs.

The BCGR program for background intensity elimination is based on our own algorithm. The background is interpolated with a polynomial of degree up to 6. The effect of spread in the raw background data is eliminated by means of the use of an intensity-deviation band. PATTR has a routine for choosing a band width directly on a display. BCGR can create output files containing a list of Bragg peak regions, refining polynomial coefficients, running protocol, and other useful information. The program runs in both autopilot and interactive mode. A user has the opportunity to mark on the pattern some areas belonging to background or peaks and to exclude areas using PATTR. This improves the result when the measured pattern is not sufficiently good.

The PEFIT program allows the separation of overlapped Bragg peaks and the refinement of position, integral intensity, and FWHM of both single and overlapped peaks. This routine employs the Powel method. The list of profile functions available for description of the peak shape includes Gaussian, two Lorentzians, pseudo-Voigt and Pearson VII. Each Bragg reflection is approximated by the $K_{\alpha}1$-$K_{\alpha}2$ doublet; the ratio of the intensities of its components can be set by the user (the default is 2:1).

The COMP utility program allows the comparison of two measured patterns, to shift them with respect to each other, and to display the difference diagram on a separate panel.

P3.4 PDN—PROGRAM FOR RIETVELD ANALYSIS, A. B. Tovbis and Ya. E. Cherner*, Institute of Crystallography, 59 Leninsky Ave., Moscow, 117333, * Institute of Physics, 194 Stachki Ave., Rostov-on-Don, 344104, Russia.

PDR IS a program for Rietveld analysis of X-ray and neutron powder diffraction patterns. It runs on IBM PCs and compatibles (540 kB memory required). The raw pattern file can contain up to 10,000 data Points, the allowed number of Bragg reflections is less than 4,000, and up to 10 phases can be refined simultaneously. The list of profile-shape functions includes Gaussian, three Lorentzians, pseudo-Voigt, Pearson VII, and Thomson-Cox-Hastings pseudo-Voigt. PDR takes automatic account of symmetry dependent atomic coordinates, anisotropic temperature factors and unit-cell parameters. The program has an extremely easy and flexible way of creating and editing input parameter flies in an interactive manner and a routine for set-up of the input files using a data entry screen (for beginners).

A color graphics utility enables the display and plotting of the raw and refined patterns, the difference diagram and a structure model.

The PDR's runtime for a test data set is half the corresponding runtime of the well known program DBW 4.1 (890101).

The use of PDR together with PD-ASSISTANT and PATCOM software packages provides the best starting model and an optimal strategy for the Rietveld refinement.

P3.5 A VERSATILE PROGRAM FOR RIETVELD REFINEMENT. J.F. Bérar(+*) and P. Garnier(+), (+) Chimie-Physique du Solide (UA 453 CNRS), Ecole Centrale, 92295 Châtenay, France and (*)LURE, Université Paris-Sud, 91405 Orsay, France.

The XND program for crystal structure refinement of powder data was written in order to take into account the correlations between data obtained in real-time experiments for which the statistical qnality of each diagram is very low.

Line profiles are represented by Voigt functions, new terms have been added to describe the evolution of line width and asymmetry. Preferred orientation effects are implemented using spherical harmonic functions. These functions are also used to allow an anisotropic broadening due to size or strain effect for the sample dependent part of the line width.

This allows us to reach very good profile agreements even if data have been collected with a conventional X-ray diffractometer ($R_{wp} = 0.035, GoF = 1.2$ have been obtained with various MV_2O_6 samples).

Some other possibilities (anomalous scattering, bond length penalties and rigid bodies) allow its use for complex structural problem : for example zeolites or stearate salts.

P3.6 A RIETVELD PC-PROGRAM SYSTEM ADAPTED FOR THE GUINIER METHOD. N. O. Ersson, Solid State Chemistry, Uppsala University, Box 531, S-751 21 Uppsala, Sweden

The incomparable sharpness of the diffraction lines obtained with a Guinier film camera may give problems when the observed data, as obtained with a film scanner,[1] are used in a Rietveld program. The problems in finding a profile function describing the complex angle dependence are well known.[2] Another complication is the deviation from linear relationship between film distance and Bragg angle that is always present on a film. Even small distortions may cause an incomplete overlap when observed and calculated data are compared.

The use of an internal calibration substance can eliminate the latter complication. We use high-purity Si, the NIST standard SRM 640b. In the presented system an angular correction function is first calculated. This function is then used to "rubber-band stretch" the theta-scale, so that the distance between two observed points is no longer constant. In practice this is obtained by interpolating the observed intensities and maintaining a constant step-length for the input data to the Rietveld refinement.

The present Rietveld program is a modified version of DBWS-9006PC written by Young and Satthivel.[3] Besides some changes in data input (to provide for different formats) functions for Lorentz factors for the Guinier geometry have been incorporated. A display program for easy graphics control of the output has also been written using the QuickBasic language, as for the correction programs. The Rietveld program is still written entirely in FORTRAN-77 language.

[1]Johansson, K.-E., Palm, T., and Werner, P. E., Rev. Sci. Instrum. 13, 1289-1291 (1980).
[2]Ersson, N.0., J. Appl.Crystallogr. 12, 295-302 (1979).
[3]Sakthivel, A., Young, R. A., User's Guide to program DBWS-9006PC (1991).

P3.7 MACINTOSH POWDER DIFFRACTION SOFTWARE.

A. W. Hewat, Institut Laue-Langevin, 156X Grenoble, 38042 FRANCE.

Personal computers now rival workstations for processing speed, and have the advantage of a common, user-friendly windowing interface. For example, the new Macintosh Quadra machine, using the 20MIPS 68040 processor, runs powder profile refinement programmes at typically 10-20 seconds per refinement cycle, rivalling the ILL-Vax central computer !

Standard mainframe Fortran, Pascal and C programmes can be compiled and run as easily as on any other computer, since there are no limitations to the size of the code, and a simple windowing environment is added automatically.

With a little work, more sophisticated windowing environments can be included, greatly simplifying the input of data and the examination of results. An example of this second class of programme is Mac-Lazy for the calculation of powder patterns. Much of the data can be entered simply by pointing and clicking, and the results plotted in independent graphic windows. The structure can be drawn in another window, rotated, scaled and 'animated' in real time to emphasise its 3D nature.

Personal computers can be justified if only for 'word-processing', but can in practice be used for much more. Macintosh versions of some of the 'classical' powder diffraction software will be shown, together with applications using more sophisticated user interfaces, such as Mac-Lazy.

P3.8 COMPUTER PROGRAM "CUVFIT" FOR DECOMPOSITION OF MULTIPHASE POWDER DIFFRACTION PATTERNS.

S. L. Wang, Department of Chemistry, National Tsing Hua University, Hsinchu, Taiwan 30043, R. O. C.

The program CUVFIT has been developed for analyzing powder diffraction patterns which may contain several phases. Determination of the intensities of completely overlapping reflections is feasible by using this program. Up to ten peaks can be fitted and refined simultaneously, including phases that can be stipulated to have only certain reflections. The program handles various peak shapes such as Gaussian, Lorentzian, pseudo-Voigt, and Pearson VII. Angular dependence of the peak's FWHM for each phase can be best-determined by the program and used in the next fitting cycle. Either shifted Tchebychev polynomials (degree 2) or the usual tangent function can be chosen for describing the behavior of the width. Other parameters include peak position, asymmetry factor, and polynomial background. The changes, such as the zero offset of the $2-\theta$ value, and constraints between various parameters, can be made easily. The program is essentially an individual profile-fitting one which aims at apportioning observed intensities to the individual components analytically. Details of the program and its applications to several complex systems will be presented.

P3.9 EXTRACTING STRUCTURE FACTORS FROM POWDER DIFFRACTION DATA BY ITERATING FULL PATTERN PROFILE FITTING.

A. Le Bail. Laboratoire des Fluorures, Université du Maine, 72017 Le Mans Cedex, France.

New strategies using old formulae sometimes lead to efficient algorithms. Every user of the Rietveld method has worked with the so-called 'Iobs' (or '|Fobs|') either by the simple presentation of the Bragg R factor or by Fourier difference calculation or so on. Some special uses of the Rietveld's formula for '|Fobs|' extraction were made (to be classified among the two-stages alternatives to the Rietveld method), however the starting $|F|$s were always the $|Fcalc|$ corresponding to a (complete or partial) structure model (Toraya, Marumo and Yamase, Acta Cryst., 1984, B40, 145-150 ; Taylor, Miller and Bibby, Z. Kristallogr., 1986, 176, 183-192; Taylor, Z. Kristallogr., 1987, 181, 151-160).
In the original Rietveld's work, the '|Fobs|' are estimated by partitioning among reflections contributing to each profile point of the pattern according to the $|Fcalc|$, thus they are biased. However, it will be shown that iterating the Rietveld's formula provides a nice and simple way to extract $|Fobs|$ as accurately as possible, starting from initial $|F|$s arbitrarily set to have the same value. The first study applying such an algorithm for structure determination purpose (using the unpublished program ARITB) was from Le Bail, Duroy and Fourquet, Mater. Res. Bull., 1988, 23, 447-452. Near 20 *ab initio* structure determinations have been now performed in this way, either from conventional or synchrotron X-ray powder data. The algorithm has been implemented as an option in two other programs (FULLPROF, GSAS); any Rietveld program can be easily modified to accommodate this possibility to extract structure factors. The strategy necessary for success will be exposed. The best results are from cell and space group constrained conditions: only all equal starting $|F|$s can ensure that the structure factors of strictly overlapping reflections are undifferentiated: they keep the same value after each iteration; it is strongly recommended to start from very precise cell parameters (not approximated ones) in order to avoid false minima in case of complicated patterns with strong overlapping: some iterations are necessary without modifying cell and profile parameters in order to start their least-squares refinement at further cycles in good conditions. A definite advantage of the procedure is the very low number of parameters to be refined (15 maximum in ARITB) allowing it to handle problems of any size. Extracting in one run the several thousand $|F|$s (for instance from synchrotron powder data with 0.02°2θ-FWHM) implicit for large and/or low symmetrical cells is not a utopia.

P3.10 MAXIMUM ENTROPY METHOD ANALYSIS OF X-RAY AND NEUTRON POWDER DIFFRACTION DATA.

M. Sakata, T. Uno, M. Takata, Department of Applied Physics, Nagoya University, Nagoya, Japan; and C. J. Howard, Australian Nuclear Science & Technology Organisation, Lucas Heights, NSW 2234, Australia

It is often said that X-ray and neutron diffraction are complementary. In X-ray powder diffraction, X-ray photons are scattered by electrons, while neutron beams are diffracted by nuclei in neutron powder diffraction, assuming that there are no magnetic interactions. What can be observed from X-ray and neutron powder diffraction data is, therefore, the electron and nuclear density distribution of the crystalline materials, respectively. It is, however, not known how to restore such electron and/or nuclear density distributions directly from the observed structure factors by powder diffraction without using a structural model.

Recently Sakata & Sato [M. Sakata & M. Sato (1990) Acta Cryst. A46, 263] have successfully applied the Maximum Entropy Method (MEM) to restore the precise electron density distribution from structure factors accurately determined by the Pendellösung method. It is possible to measure accurate Bragg intensities free from extinction effects by a powder diffraction experiment. In this study, the MEM analysis of both X-ray and neutron powder diffraction data will be given. In the neutron diffraction case, it was necessary to overcome the difficulty of negative scattering length of some atoms, such as H, Ti, Mn. Both the electron and nuclear density distributions of rutile were obtained from X-ray and neutron diffraction data. In the electron density distribution, apical and equatorial bonds of rutile are clearly shown, while it is shown in the nuclear density distribution that the nuclei of Ti and O are located within very confined regions around atomic sites.

P3.11   A BAYESIAN APPROACH TO BRAGG PEAK LOCATION IN POWDER DIFFRACTION PATTERNS. W.I.F.David, R.M.Ibberson and D.S.Sivia, ISIS Facility, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon., OX11 0QX, U.K.

This paper presents an ab-initio Bayesian approach to Bragg peak location in powder diffraction patterns. It addresses the question: "Given that a region of a powder diffraction pattern is presumed to contain a few Bragg peaks and that the instrumental resolution function is known, what is the most probable number of peaks and what are their positions and intensities?". One of the major advantages of this Bayesian approach is that the relative probabilities of the number of possible peaks is quantified. Two applications are illustrated: (i) the location of Bragg peak positions in an unknown powder diffraction pattern prior to auto-indexing and (ii) the unit cell determination of pseudo-symmetrical structures.

P3.13   THE EFFECT OF THE THIRD ANGULAR PARAMETER IN TWO-CIRCLE DIFFRACTOMETRY. Cong Qiuzi, Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, People's Rep. China

On the basis of the θ-and 2θ-angles in a two-circle diffractometer, a third α-angle between the sample surface and the incident X-ray beam is introduced, so that three X-ray scan modes of $\theta/2\theta$, 2θ and θ are normalized into a common form of $\theta(\alpha)/2\theta$. Subsequently, based on the asymmetrical Bragg reflection geometry, equations of the diffraction intensities and the observed crystal-plane azimuthal angles are derived as the following common forms;

$$I = I(\alpha,2\theta,t) \cdot P \quad (1) \qquad \text{and,} \quad \Phi_{hkl} = \theta_{hkl} - \alpha \quad (2)$$

where t is the film thickness or effective X-ray penetration depth, P is the reflection profile function, and θ_{hkl} is the Bragg angle, for the given reflection.

Changing one or two parameters in eq. 1 we can obtain three groups of equations corresponding to three scan modes respectively.

a. For $\theta(\theta)$ / 2θ (called CBD) scan mode,
$$Ic = Ic(\theta,2\theta,t) \cdot Pc \quad (3) \qquad \text{and,} \quad (\theta_{hkl})c = 0° \quad (4)$$

b. For $\theta(\alpha_o)$ /2θ (called STD) scan mode,
$$Is = Is(\alpha_o,2\theta,t) \cdot Ps \quad (5) \qquad \text{and,} \quad (\Phi_{hkl})s = \theta_{hkl}-\alpha_o \quad (6)$$
where α_o is an angular parameter in the range from 0.6° to θ_{hkl} for Cu K_α radiation.

c. For $\theta(\theta)/2\theta_{hkl}$ (used for ADA) scan mode,
$$Ta = Ta(\theta,2\theta_{hkl},t) \cdot Pa \quad (7) \qquad \text{and,} \quad (\Phi_{hkl})a = \theta_{hkl}-\theta \quad (8)$$
where θ is the rotating angle of the specimen, and $2\theta_{hkl}$ is the diffraction angle for the given (hkl) reflection.

The effect of each scan mode in X-ray diffractometry is well known, for example, the "a" mode is most used to determine phases, as is the "b" mode. Furthermore, the effective X-ray penetration depth can be controlled with "b" mode by adjusting the sample-tilting α_o-angle; at the same time, the film-layer or film thickness can be fitted with a combination of "c" and "b" modes.

P3.12   TREOR90: AN IMPROVED VERSION OF THE TRIAL-AND-ERROR INDEXING PROGRAM TREOR. L. Eriksson, P.-E. Werner, and M. Westdahl, Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-106 91 Stockholm, Sweden.

Although high quality data is the most important prerequisite for powder indexing, the rate of success of a semi-exhaustive program for powder indexing is most strongly dependent on the built-in crystallographic experience. Accumulated experience from the use of the trial-and-error indexing program TREOR[1,2] has been incorporated in the new version, TREOR90. Dominant zone tests, originally used only for the monoclinic symmetry have been added for the orthorhombic and triclinic symmetries. Dominant zone tests are made prior to the conventional trials, thus preventing the most frequently occurring situation when figure of merit tests in powder indexing may fail.

Other new features included in the program are: successive reduction of trial cell volumes by statistical criteria, automatic unit cell reduction and conversion to conventional cells according to the metric symmetry, a condensed output file and an increased over-all efficiency of the indexing algorithms.

The original key-word system is retained, but at the expense of a relatively small increase in computing time the user may normally accept a default setting of parameters to run all symmetries from cubic to triclinic.

Computing times by the vectorized TREOR90 for triclinic and monoclinic patterns have never exceeded 5 and 2 minutes, respectively, on a CONVEX 210, which is about 5% of the time required on a Micro-VAX II.

[1]P.-E. Werner, Z. Kristallogr., 120, 375-387, 1964.
[2]P.-E. Werner, L. Eriksson and M. Westdahl,J. Appl. Crystallogr. 18, 367-370, 1985.

P3.14   RIFRAN '92: THE IBM PC SYSTEM FOR AUTOMATIC X-RAY POWDER DIFFRACTION PHASE ANALYSIS. T. Havlik, M. Skrobian, and F. Petricko, Technical University of Kosice, Czechoslovakia.

An efficient identification system RIFRAN '92 was developed for rapid qualitative and quantitative X-ray powder diffraction phase analysis of polycrystalline materials. The system is represented by a set of interactive computer programs written in the Borland Turbo C++ and Borland Turbo Pascal 6.0 computer languages and implemented on the IBM PC computer working under MS DOS.

The identification procedure for the analysis is based on comparison of the diffraction patterns of standards with those of the unknown sample, utilizing statistical criteria and chemical analysis for qualitative analysis, and the method of mathematical optimization for quantitative analysis.

The JCPDS database is used for qualitative analysis, as well as the specialized database for quantitative analysis in which the standards were calculated previously[1].

The system enables the specialized database preparation according to the requirements of the user, using chemical composition, mineralogical name, subfiles, PDF numbers, etc. Thus, the analysis is more effective and faster. Specification of the chemical criteria with the aid of Boolean operands is used to further enhance the procedure.

The analysis takes only a few seconds in the average case. The results are sorted according to statistical criteria, or chemical analysis. Output of results is printed on the screen, as well as to the printer or a file. Both numerical and graphical output of the results are possible.

[1]Smrcok L., Weiss Z.: J. Appl. Cryst.—in press.

P4.1   NONDESTRUCTIVE RESIDUAL STRESS MAPPING BY NEUTRON AND X-RAY DIFFRACTION METHODS. C.R. Hubbard*, T.A. Dodson, S.A. David, Metals and Ceramics Division, and S.Spooner, Solid State Division, Oak Ridge National Laboratory, Oak Ridge TN 37831-6064, Phone (615) 574-4472.

Advanced X-ray and neutron diffraction facilities to map macro and micro residual stresses have been established at Oak Ridge National Laboratory. For mapping stresses within the specimen by neutron diffraction, attachments for a triple axis spectrometer at HFIR have been built and automated which include beam collimators, an XYZ-chi specimen stage, and a linear position sensitive detector. Sampling gauge volumes of 20 to as little as 2 mm^3 have been successfully used in mapping stresses in several specimens including a ferritic steel plate with a multipass weld, weld simulation test samples, and a brazed zirconia to iron joint. Results of mapping macro residual stresses in these specimens show significant stress gradients. Micro residual stresses in ceramic and metal matrix composites have been studied with both high resolution neutron powder diffraction and the triple axis spectrometer attachment. Comparison of the sensitivity and time required for measurement of microstresses will be presented. For mapping stresses at the surface a new X-ray mapping facility was developed which consists of an 18 KW rotating anode generator and a Scintag PTS goniometer. Capabilities of this system will be presented, and data collected by both neutron and x-ray method will be compared.

P4.2   ABOUT ANALYTICAL MODELS FOR TEXTURE CORRECTION. M. Järvinen, Department of Information Tecnology, Lappeenranta University of Technology, SF-53851 Lappeenranta, Finland.

Powder diffraction is commonly used for obtaining information on the properties of materials. Accurate intensity measurements are needed, for instance, for the determination of the volume fractions of phases in a sample or for refinement of the structures of different materials.

The accuracy of results depends significantly on the treatment of sample effects. Loose samples can suffer from inhomogeneity and dense samples can be bothered by preferred orientation or texture. In practice, a sample with random orientation is very difficult to prepare, so that usually dense samples are used in measurements, and the texture effects are corrected mathematically. For this procedure analytical models are needed for presentation of the orientation distribution in the sample.

Very elegant theoretical and experimental methods have been created for determination of the texture factors of a sample, but often these methods are too complicated and superfluous for practical use. That is why simplified methods have been developed for some frequently occuring cases, for instance, a Gaussian function or March function is used for the description of fiber texture.

In the present paper the use of Gaussian and March functions are compared with the use of symmetrized harmonics by making model calculations and analyzing Ni and Mg powder data. The accuracy of these analysis methods are evaluated, and the advantages and the limitations of different models are discussed.

P4.3   SAMPLE-INDUCED ERRORS IN QUANTITATIVE TEXTURE ANALYSIS FROM DIFFRACTION POLE FIGURES. A. Mücklich and P. Klimanek, Institute of Ion Beam Physics and Materials Research, Research Centre Rossendorf, DO-8051 Dresden, Germany, and Institute of Metal Science, Mining Academy Freiberg, DO-9201 Freiberg/Sa., Germany

During the last decade many efforts have been made to improve the reproduction of orientation distribution functions (Odf) of polycrystalline materials from X-ray and neutron diffraction pole figures. The advances achieved in this field are in contrast with a relatively unsatisfactory level of data evaluation due to the neglect of a number of systematic errors, which can significantly influence the reliability of quantitative texture analysis. In this connection two types of uncertainties have to be taken into account:

- experimental errors which, however, can be kept sufficiently small by suitable choice of the conditions of investigation, and

- sample-induced errors caused by the microscopical architecture (e.g. lattice perfection of the crystallites, grain size and orientation distribution) of the material taken under investigation.

Errors of the second type are characterized by the fact that they modify the actual absorption and/or scattering behaviour of the sample material. This is particularly important in investigations of structurally inhomogeneous polycrystalline materials [P. Klimanek in: X-Ray and Neutron Structure Analysis in Materials Science (Ed. J. Hašek).Plenum Press, New York 1989, 125; EPDIC 1 - European Powder Diffraction (Ed. R. Delhez, E.J. Mittemeijer), Mater. Sci. Forum, Vol. 79-82, Pt.1, 73. Trans Tech Publ., Aedermannsdorf/Switzerland 1991]. After a brief discussion of general aspects of pole figure evaluation, the present paper considers three effects that give rise to errors of the second type in the case of neutron diffraction:

1. absorption weighting of diffraction pole figures from polycrystalline layer structures with texture gradients or different texture components at various distances from the sample surface,

2. structure-induced extinction in materials whose texture components clearly differ in grain size and/or lattice perfection of the crystallites [A. Mücklich, P. Klimanek: Mater. Sci. Forum Vols. 79-82 (1991) 185] and

3. texture-induced secondary extinction in polycrystals with strong preferred orientation [A. Mücklich, P. Klimanek in: Cryst. Res. Technol. 23 (1988) K105; Mater. Sci. Forum Vols. 70-82 (1991) 185].

The practical importance of the effects is illustrated by computer simulation of neutron diffraction pole figures as well as by experimental results obtained with copper and iron.

P4.4   CHARACTERIZATION OF PREFERRED ORIENTATION IN CRYSTALLINE MATERIALS BY X-RAY POWDER DIFFRACTION WITH THE MARCH FORMULA APPRAISAL OF THE LINE RATIO METHOD. H. Sitepu, Centre of Basic Sciences, Indonesian Institute of Technology, Serpong - Tangerang, Indonesia. 15320; B. H. O'Connor and Li Deyu, Department of Applied Physics/Centre for Materials Technology Curtin University of Technology, Perth, Australia.

Texture, i.e. preferred orientation, can cause large systematic errors in quantitative analysis of crystalline materials using x-ray powder diffraction (XRPD) data. Various mathematical forms have been proposed for the application of preferred orientation corrections. The most promising of these appears to be the single-parameter March (1932) model proposed by Dollase (1986).

Li et al. (1990) and O'Connor et al. (1991) applied the March model to determine the level of preferred orientation in various gibbsites using two procedures. The first involved the Rietveld (1969) least squares pattern fitting method. Each pattern was Rietveld-analyzed in two ways, initially assuming random orientation of the crystallites and subsequently with the March model. The second procedure for preferred orientation analysis, described here as the line ratio method, determines preferred orientation factors according to the intensity ratios of carefully selected line pairs.

In this study the procedures proposed by Li et al. and O'Connor et al. for texture analysis have been evaluated with XRPD data sets for molybdite, calcite and kaolinite. The results indicate that, while the March formula improves agreement between the calculated and measured patterns in Rietveld analysis, other forms of systematic error in the intensity data appear to limit the effectiveness of the March formula in general. It has been found also that the line ratio method improves agreement between the data sets, but less effectively than the Rietveld method. It is proposed that extinction is likely to be the most influential source of systematic error competing with texture.

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Rietveld, H. M., 1969, J. Appl. Cryst., 2:151.

P4.5 MICROSTRUCTURAL EFFECT ON THE CORROSION OF TUNGSTEN IN SF$_6$. W. Wong-Ng, L. P. Cook, and C. S. Choi, Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, U.S.A.

Stored chemical energy release systems based on liquid metal combustion in general require the injection of a gaseous oxidant through a nozzle of chemically resistant material. Tungsten (W) has been the traditional metal of choice. Understanding of the kinetics of reaction of W with the oxidant SF$_6$ as a function of temperature is of primary importance for predicting service lifetime. Scanning electron microscopy study of tungsten rods manufactured by a swaging process and exposed to SF$_6$ indicated a significant orientational effect on the corrosion process, possibly originating from the considerable granular elongation parallel to the rod axis produced during the fabrication process. X-ray analysis of these rods showed preferred orientation. Texture measurement was subsequently conducted using neutron diffraction. This paper discusses the result of the pole figure study and the correlation of the relative rate of reaction with the grain alignment.

P4.6 FITTING X-RAY PROFILES IN TERMS OF PHYSICALLY RECOGNIZABLE PARAMETERS. R.W. Cheary and A. Coelho. Department of Applied Physics, University of Technology Sydney. PO Box 123. Broadway, NSW. Australia 2007.

X-ray powder line profiles are generated by convoluting the instrumental aberration functions of a Bragg-Brentano diffractometer with the Cu Kα emission spectrum and the diffraction function arising from the presence of either small crystallites or strain in the specimen. These profiles are fitted to experimental profiles using a combination of multilinear regression and Newton-Raphson non-linear least squares fitting based on numerical differentials. In addition to the 2θ values and integrated intensities of the lines. the fitted instrumental parameters include the receiving slit width, the length of the receiving slit, the x-ray target width, the divergence angle of the incident beam and a "top hat" mis-alignment function which can be used to represent defocussing effects or specimen tilt. The wavelengths and lifetime widths of the five components of the emission spectrum are fixed by the spectrometric measurements of Berger(1). The relative intensities of these components are calibrated with an Si crystal or a standard 2μm polycrystalline MgO disc to allow for differential attenuation by either a diffracted beam or incident beam monochromator. Crystallite size effects are assumed to produce a Lorentzian profile whilst strain produces a Gaussian shape: the refined parameters are the apparent crystallite size and the RMS strain. In practice each instrumental parameter is constrained to be the same for all profiles from a particular specimen. As some of the parameters are either highly correlated with one another or do not contribute significantly to the profile shape. it is only possible to refine on two or three instrumental parameters even when diffraction data over a wide range of 2θ values are being fitted. Refining on all the instrumental parameters simultaneously normally produces results with little physical validity. The successful application of this convolution fitting procedure to a standard powder diffractometer has already been discussed(2). In this work we will demonstrate how the fitting procedure can be used for standardless line profile analysis and the measurement of crystallite size and stacking fault defects. We will also examine the consistency of the fitted instrumental parameters when applied to two additional powder diffractometer configurations. In the first case the effects of incorporating a high resolution Ge monochromator (CuKα₁only) in the incident beam are considered. In the second system, the effects of replacing the receiving slit/detector with a position sensitive detector are considered and how the broadening of the line profile under non-focussing conditions is incorporated into the fitting procedure.

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P4.7 INSTRUMENTAL BROADENING DETERMINATION FOR XRD PROFILE ANALYSIS. P. Scardi & L. Lutterotti, Dipartimento di Ingegneria dei Materiali, Universitá di Trento, 38050 Mesiano (TN), Italy; Paul Maistrelli, Dipartimento di Fisica, Universitá di Trento, 38050 Povo (TN), Italy.

A standard sample for XRD instrumental profile determination was prepared by pressing -32+45μm KCl powder to obtain a tablet. After a suitable annealing treatment, the tablet gave very sharp profiles (FWHM=0.07° and 0.04° at $2\theta = 28.4°$ for the normal and high resolution set-up, respectively). The experimental patterns were modelled by non-linear least squares fitting using pseudo-Voigt (pV) or Pearson VII (PVII) functions, corrected for asymmetry, and the trend of fitting parameters describing the curves (ω and η for pV, ω and m for PVII) as a function of 2θ was measured. These trends, which can be described by fitting data with a low degree polynomial, can be used for a fast and reliable 2θ dependent instrumental correction in line broadening analysis, both using raw experimental data and modelled profiles.

The features of the experimentally determined instrumental function were studied using several diffractometers, always in Bragg-Brentano geometry, with different set-ups. In particular, the effect of using a monochromator in the diffracted beam and changing the slits width was studied. It was found that the goniometer radius, beside having a remarkable influence on the resolution (peak width) of the diffractometer, also affects low angle peak asymmetry. High resolution geometry (narrow slits and large radius) is highly advisable if sharp and fully symmetric profiles are needed, especially at low angles. In spite of the loss in intensity, involving an increase in the data collection time necessary to obtain a given precision, this kind of set up can greatly enhance the limit for crystallite size and microstrain determination through line broadening analysis. Also, structure refinements from X-ray powder diffraction data give better results when the instrumental peak is sharp and symmetric.

P4.8 A PREDICTABLE DIFFRACTION PATTERN - METHOD AND SUBSTANCE. V. A. Kogan, Physical Dept. of Rostov State University, Zorge St. 5, Rostov-on-Don, 344104, Russia

The purpose of this work is to study the possibility that X-ray powder diffraction profiles can be computed independently. The problem may be divided into two parts:

i) development of the method of computation

ii) synthesis of a stable substance in which the factors of physical broadening are small and may be described on an unempirical scale.

The Fourier-synthesis method [Kogan & Kupriyanov, 1992] implemented in the computer program for full profile refinement, KET, was used to compute the reflection profiles theoretically. This programme makes it possible to synthesize powder patterns for Bragg-Brentano geometry by taking into account the dimensions of the geometrical system, the spectral distribution, porosity effects and a model for imperfections in the sample. The 5μm fraction of strontium titanate was used as a sample. The crystallites obtained were of cubic habit and were not subjected to any external influences after synthesizing. It was assumed that the coherent scattering regions coincide with the geometrical crystallite sizes, and that micro-deformations of the crystal lattice are absent. During the experiments the diffraction profiles were recorded over a wide angular range for two different geometrical conditions. Full-profile refinement without introducing varying 'profile' parameters (R_{prof}=.05-.07) provided adequate values of the structure parameters in different experiments.

References

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P4.9   X-RAY TRACING, A TOOL FOR IMPROVED ACCURACY IN POWDER DIFFRACTOMETRY

J. Timmers (Philips Research Laboratories, P.O. Box 80.000, NL-5600 JA Eindhoven, The Netherlands), R. Delhez, F. Tuinstra (Delft University of Technology, The Netherlands), F. Peerdeman (Philips IE Analytical, Almelo, The Netherlands)

The dimensions of X-ray optical components as source, specimen and slits have a significant influence on the positions and shapes of powder diffraction lines and establish a well-known source of error (the so-called geometrical aberrations) in powder diffraction analysis. With the advancing mechanical goniometer accuracy in mind, corrections for geometrical aberrations, in order to arrive at improved accuracy of peak positions and improved interpretation of line shapes, become increasingly relevant.

The majority of geometrical aberration sources can be quantified analytically (see e.g. A.J.C. Wilson, Mathematical Theory of X-ray Powder Diffractometry, Eindhoven Centrex 1963) in terms of centroid shift and variance of the geometrical part of the instrumental line profile. However, for the full geometrical line profile neither the shift of the peak maximum nor the shape can be obtained analytically. Therefore, a versatile computer program has been developed in order to simulate X-ray powder line profiles from the known dimensions of the diffractometer and its optical components. Diffraction geometries to be simulated include the Bragg-Brentano, Seemann-Bohlin and Schulz (texture) geometries as well as all non-focusing geometries.

As an example, peak shifts have been calculated for two Philips diffractometer systems: the Integrated Powder Diffractometer (PW1800) and the new X'Pert diffractometer. Simulated and measured peak shifts for the NBS silicon powder standard (SRM640b) are compared in figure 1. A clear discrepancy exists between peak and centroid shifts in the low-angle region.
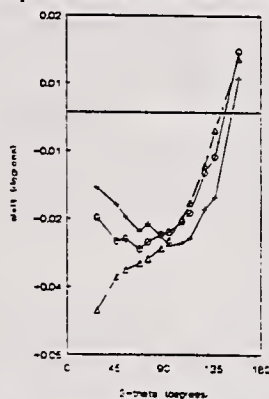

Figure 1. Numerically calculated peak (-0-) and centroid (-△-) shifts as well as measured peak shifts (-+-) as a function of Bragg angle for the NBS silicon standard SRM640b. All data refer to the Philips Integrated Powder Diffractometer.

P4.10   ACCURACY IN CRYSTALLINITY DETERMINATION BY MEANS OF X-RAY POWER DIFFRACTOMETRY.

G. Berti, Dipartimento Di Scienze Lella Terra - Univ di Pisa -Via S. Maria 53 - 56100 Pisa - Italy

According to the symmetry rules of the crystalline system used to describe the crystallographic features of the sample, the crystallinity may be considered as a natural performance of the ideal crystalline state. The crystallinity is a feature pertaining to the sample, not to the crystalline system. The achievement of a high level performance is ruled by the crystallization conditions which act on the crystal evolution and produce mosaicity and also other observable results. Unfortunately, the effects related to the compositional variation, phase transformation, and planar defectivity are not directly visible on the diffraction pattern. They have to be deduced by reconstructing both the reciprocal average lattice and the shape of the nodes. The starting point of the reconstruction is the profile fitting of the experimental pattern by means of suitable analytical functions. The accurate determinations of positions and shapes of the peaks in the powder pattern are useful to find the proper lattices and shapes of nodes. So the crystallinity determination requires a phenomenological description as accurate as possible where, besides the many instrumental aberrations, crystallinity and structure factors are taken into account. To this aim the mathematical theory of X-ray powder diffraction suggests simple and effective relationships.

Some samples of quartz show that the crystallinity and the crystallite volume are closely related when an equivalent average lattice is used to describe the assemblage of defective and/or disordered cells.

P4.11   X-RAY PROFILE ANALYSIS IN STRUCTURALLY INHOMOGENEOUS POLYCRYSTALS.

P. Klimanek, Department of Materials Science, Academy of Mining, P.O.B. 47, DO – 9200 Freiberg/Sa., Germany

X-ray diffraction peaks of real polycrystals as, for instance, bulk materials or real powders interesting in materials science and technology have, in general, to be considered as diffraction multiplets (i.e. as weighted sums of partial reflections) due to the volume fractions of lattice disorder-statistics that are formed by crystallite groups with significantly different degrees of structural perfection. Accordingly the macroscopically observable line broadening of such reflections must be interpreted in terms of a generalized sample statistics representing the microscopical architecture of the scattering crystallite ensemble, which encloses

1. the type of the lattice-disorder statistics, and

2. the mean grain size, the orientation distribution (texture), and the spatial arrangement of the structurally different crystallite groups.

After a brief representation of the formalism needed in this case (Klimanek,P. in: Freiberger Forschungsheft B 265 (1988) 76; X-Ray and Neutron Structure Analysis in Materials Science (Ed. J.Hasek). Plenum Press, New York 1989, 125; EPDIC 1 - European Powder Diffraction (Ed. R.Delhez, E.J.Mittemeijer), Mater. Sci. Forum, Vol. 79-82, Pt.1, 73. Trans Tech Publ., Aedermannsdorf/Switzerland 1991) some important methodological aspects of the analysis of X-ray multiplet profiles are discussed and illustrated by experimental results obtained with model specimens (powders) and real polycrystalline materials.

P4.12   A STUDY OF CRYSTALLITE SIZE AND STACKING FAULTS IN EX-OXALATE ZnO BY PATTERN DECOMPOSITION.

D. Louër*, A. Boultif*, J. P. Auffrédic* and J. I. Langford† *Laboratoire de Cristallochimie, Université de Rennes I, Avenue du Général Leclerc, 35042 Rennes cedex, France, and †School of Physics ans Space Research, University of Birmingham, Birmingham B15 2TT, U.K.

Previous X-ray diffraction studies of ex-hydroxide nitrate powder ZnO demonstrated that the form of the crystallites was prismatic, on average approximating to cylinders (Louër et al., J. Appl. Cryst. 16, 183-191, 1983). Recently, data were obtained for the first 27 lines of the diffraction pattern for ZnO obtained from thermal decomposition of the oxalate, by using the same sample as had been previously investigated by means of electron microscopy and adsorption calorimetry (Bolis et al., J. Chem. Soc. Faraday Trans. 85, 855-867, 1989). Integral breadths obtained by total pattern fitting again have a marked hkl dependence; an indexed Williamson-Hall plot indicated that microstrains are negligible and that there are three groups of reflections, according to the values of hkl. This is a classic example of the effects of stacking faults combined with broadening due to crystallite size and it is the first time that the former has been reported for ZnO.

Twelve reflections with $l=0$ or $h-k=3n$ are unaffected by structural 'mistakes' and these were used to obtain mean crystallite dimensions, based on spherical and cylindrical morphologies. From the dimensions obtained [$<D>=453(17)$ Å for spheres and $<D>=404(7)$ Å, $<H>=351(9)$ Å for cylinders)], the 'size' contribution to the remaining reflections ($h-k=3n \pm 1$) can be calculated. This can then be separated from the total breadth for these lines to obtain the 'mistake' component and hence the stacking-fault probability. For this purpose it is customary to assume that the constituent line profiles are Lorentzian (eg. Warren, X-ray diffraction, 1969, p. 303), but here all sample line profiles, including those broadened solely by 'size' effects, have an appreciable Gaussian component. The Voigt function may therefore be a better approximation to the 'size' profiles and an alternative analysis based on this function (eg. Langford, J. Appl. Cryst. 11, 10-14, 1978) was also carried out. From the Lorentzian model and cylindrical form, the mean probability of the occurrence of a stacking fault was found to 0.007(3), corresponding to a separation between fault planes approximately equal to the mean height of the crystallites. By assuming Voigtian 'size' profiles, the probability was about 0.014(9), equivalent to ~1 fault per crystallite, on average.

This study has also demonstrated that with high quality data, detailed self-consistent and fairly precise information about structural imperfections can be obtained from a combination of line-profile analysis and total pattern fitting. The ex-oxalate ZnO analysis represents a fairly demanding case, since the fault probability is small and the crystallites are relatively large, and a significant factor in obtaining such detailed information was the use of a high resolution diffractometer. On the other hand, it revealed some of the limitations of the procedure used to model the diffraction line profiles.

P4.13   DETERMINATION OF CRYSTALLITE-SIZE AND LATTICE-STRAIN PARAMETERS FROM X-RAY DIFFRACTION LINE-PROFILE ANALYSIS BY APPROXIMATING BOTH SIZE AND STRAIN BROADENED PROFILES WITH THE VOIGT FUNCTION. D. Balzar[*] and H. Ledbetter, Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Boulder, Colorado 80303, U.S.A.

When the pure-specimen (physical) broadened profile can be satisfactorily described with a Voigt function, by applying the Langford (1980) method, it is possible to separate exactly the size $A^S(L)$ and strain $A^D(L)$ Fourier coefficients. The column-length distribution functions are analytically derived. It is shown that $<D>_s \leq <D>_v \leq 2<D>_s$, where $<D>_s$ and $<D>_v$ refer to surface-weighted and volume-weighted average domain sizes. Distribution of average strain is evaluated. For not too large a harmonic number, mean-square strain decreases linearly with distance L, but generally a more complex relationship holds with a finite value for $L=0$. Errors are estimated, and the method is compared with the Warren-Averbach (1952) analysis and its modification (Delhez et al., 1980) for separation of size and strain parameters. Some problems with the application are described.

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Langford, J.I. (1980). *ibid.*, pp. 255-269.
Warren, B.E. & Averbach, B.L. (1952). *J. Appl. Phys.* 23, 497.

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[*]On leave from Department of Physics, Faculty of Metallurgy, 44000 Sisak, Croatia.

P4.14   A COMPARATIVE STUDY OF WARREN-AVERBACH, MITRA-MISRA AND de KEIJSER METHODS FOR CRYSTALLITE SIZE AND STRAIN ANALYSIS IN NiO POWDER. L.D. Pradhan and B.S. Acharya, R.R.L. Bhubaneswar-751013, Oriss, INDIA.

Out of all the methods available for analysis of crystallite size and strain in polycrystalline materials, the three most widely used methods are Warren-Averbach (WA), Mitra-Misra, and the recently proposed method of de Keijser et al. In many cases it is difficult to use the WA method because of the nonavailability of multiple orders of reflections and the fact that the shape of the profile from Cauchy or Gaussian. The method proposed by de Keijser et al. takes into account the shape of the profile function, whether it is Cauchy, Gaussian, Voigt, Pseudo Voigt, or Pearson VII. Nickel oxide powder, the material used in the present investigation, gives an interesting case study for comparison of the above three methods. This is due to the fact that multiple order reflections are available, and the shape of the profile is Voigtian. Line profile analysis of the powder has been performed repeatedly at different temperatures, including 300, 773, 873, and 973 K, and the crystallite size and strain values have been calculated by using the WA, Mitra-Misra, and de Keijser methods. In calculating strain from the de Keijser method, the Voigt, Pseudo Voigt, and Pearson VII functions have been used. It has been observed that the de Keijser method does not yield meaningful results, whereas the WA and Mitra-Misra methods do give good outcomes. The cause of this variation is not known. However, it is tentatively recommended that even if the profiles fit the equations for Voigt, Pseudo Voigt, or Pearson VII, one should perform all the above analytical methods before arriving at any conclusion.

P4.15   X-RAY DETERMINATION OF THE CRYSTALLITE-SIZE DISTRIBUTION FUNCTION FOR ALKOXY-DERIVED BARIUM TITANATE POWDER MATERIALS. V. V. Zhurov, S. A. Ivanov, M. I. Yanovskaya, V. K. Lymar', Karpov' Institute of Physical Chemistry, 103064 Moscow K-64, Russia.

A detailed knowledge of the crystallite-size distribution function (CSDF) is required in many fields of materials science. A careful analysis of X-ray diffraction line broadening provides a convenient method for obtaining this information. The purpose of our work was to determine the CSDF in barium titanate powders prepared by hydrolysis is of alkoxides with or without further heat treatments.

X-ray powder diffraction experiments (URD-63 diffractometer, Cu-radiation, T = 300 K) were combined with electron microscopy (JEM-1000X) and analysis with Fritsoh laser partical sizer. Some peaks in the diffraction patterns of our samples were carefully recorded by the step-scanning technique. The instrumental and spectral broadening was obtained from a single crystal standard. The regularization method of Fourier X-ray line profile analysis was applied for the CSDF determinations. All calculations were performed by special programs adapted to an IBM-PC computer. Some of the CSDF curves obtained are shown in the figure. There is a quite reasonable agreement between the sizes obtained from X-ray profile analysis and other physical methods up to 1 μm. The particles above 1 μm detected by electron microscopy are not monocrystalline. Values of crystallite size were computed for several crystallgrahic directions. The shape of crystallites is isotropic for all tested samples.
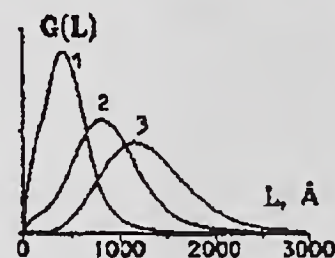
1) initial powders
2) after heating at 800°C
3) after heating at 1000°C

P4.16   X-RAY DIFFRACTION STUDY OF THE EFFECT OF SiC ON THE DEFECT CHARACTERISTICS OF Al-Mg-Zn ALLOY. R. Sen, S. K. Chattopadhyay, and S.K. Chatterjee, Regional Engineering College, Durgapur-713209, INDIA

Silicon carbide was introduced into the matrix of an Al-Mg-Zn alloy (5.98 wt% Zn, 2.91 wt% Mg, and traces of Cu, Mn, and Fe) and X-ray diffraction line profile analyses were performed to evaluate the role of SiC in the composite matrix. Peak shift peak asymmetry and Fourier line shape analysis carried out in detail in the cold worked systems have revealed that the concentration of twin faults introduced into the matrix increases with the increase in SiC concentration (from 0 to 20 wt%). The other fault parameters, such as intrinsic and extrinsic faults, have been found to be negligible, as expected in aluminium based systems having high stacking fault energy. The observations obtained from X-ray analysis have been supported by scanning electron and optical metallographic studies.

O5.1 A HIGH RESOLUTION X-RAY POWDER DIFFRACTION AND MOLECULAR MODELLING STUDY OF THE STRUCTURAL CHANGES ACCOMPANYING ABSORPTION OF TRANS-STILBENE INTO H-ZSM-5 ZEOLITE. J. B. Parise, SUNY Stony Brook, NY 11974-2100, J. Hriljac and D. E. Cox, BNL, Upton NY11972, Dr. R. Corbin and V. Ramamurthy, DuPont Company, Wilmington De 19880-0262, USA

The restricted pore geometry available in molecular sieves makes them excellent candidate materials for a number of selective chemical reaction. These have recently included the first stabilizations of the cation radicals of the *trans*-polyenes[1]. We have studied stilbene in H- ZSM-5[1] and have suggested the molecule absorbs into the straight channels on the basis of NMR data[1]. High resolution X-ray data collected at room temperature have been modelled, using a commercial software package, and are consistent with the interpretation of the NMR data.

Sorption of *trans*-stilbene causes the monoclinic H-ZSM-5 to transform to a pseudo tetragonal phase at room temperature with no detectable distortion of the unit cell. Upon cooling to 20K a pronounced orthorhombic splitting is evident. The figure above shows scans through the (400/040) reflections from *trans*-stilbene/H-ZSM-5 at selected temperatures up to 300 K collected at a wavelength of 1.3064 Å.

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1. Ramamurthy *et al. J. Amer. Chem Soc.*, **1991**, 113, 594)
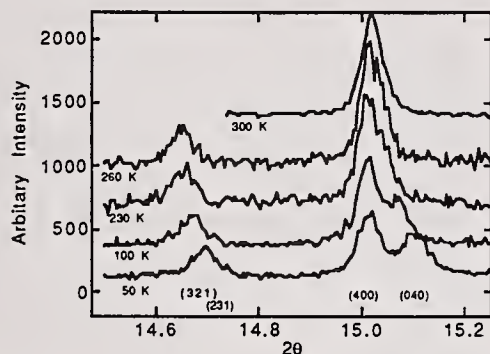
O5.2 X-RAY LINE BROADENING FROM INTERGROWTHS AND ANTIPHASE DOMAINS IN BARIUM HOLLANDITE COMPOUNDS. R.W. Cheary and R. Squadrito, Department of Applied Physics, University of Technology Sydney, PO Box 123, Broadway, NSW, Australia 2007.

Barium hollandites $Ba_x (A_yTi_{8-y}) O_{16}$ generally form over the composition range x=8/7 to x=4/3 where A is normally a 2^+ or 3^+ ion such Al, Mg or a metal from the first transition series. As the tunnel sites accommodating the Ba are only partially occupied the Ba and vacant sites usually adopt an ordered configuration along the tunnels and superlattice lines appear in the x-ray powder pattern. These compounds undergo an order-disorder transition and above the transition temperature T_c the superlattice lines disappear. For the hollandites $Ba_x (Mg_xTi_{8-x}) O_{16}$, T_c goes from 710°C at x=8/7 to 900°C at x=4/3. When these materials are first cooled below the transition temperature T_c, the superlattice lines are broadened through the formation of intergrowths and antiphase domains. On further annealing the superlattice lines become sharp and also undergo a 2θ shift. No significant changes are observed in the hollandite lattice lines on ordering. At x=4/3, Ba ions occupy 67% of the sites and the ordering sequence is Ba-Ba-V with a $3c_{holl}$ supercell where V represents a vacancy. When the occupancy drops to 60% (x=1.20) the ordering sequence is Ba-Ba-V-Ba-V with a $5c_{holl}$ supercell. At intermediate occupancies, the structure consists of intergrowths of regions with Ba-Ba-V order and Ba-Ba-V-Ba-V order. For x<1.20, the structure switches between regions of Ba-Ba-V-Ba-V order and Ba-V order. In all compositions lateral ordering occurs between tunnels. This is manifested as unbroken corrugated layers of vacancies perpendicular to the tunnels. To fully describe partially ordered hollandites it is also necessary to include antiphase domains into the model with boundaries parallel to and perpendicular to the tunnels. In this work a general diffraction theory is developed for interpreting the breadths and shifts of the superlattice lines from partially ordered polycrystalline hollandites. This will allow the kinetics of ordering to be studied through the time and temperature dependence of the superlattice line breadths. Analytical expressions for x=4/3 hollandites that only contain antiphase domains have recently been published(1). This work has now been generalised to incorporate intergrowths in the model as well as antiphase domains. In all cases the diffraction profiles are Lorentzian. Results will be presented for a range of hollandites at different Ba concentrations. These include the 2θ shifts and apparent crystallite sizes corrected for instrumental effects for different superlattice lines from both fully ordered and quenched specimens. We have been able to measure the mean dimensions of the ordered regions and identify both the boundary layer structure and the hkl planes defining the boundaries. In monoclinic hollandites the boundaries form on the 101 and 10$\bar{1}$ planes parallel to the tunnels and on the 010 perpendicular to the tunnels, but after extended annealing only 010 boundaries remain.

(1) Cheary, R.W. & Squadrito, R. (1992) Acta Cryst. A48, In Press

O5.3 A PHYSICAL MODEL FOR X-RAY ANALYSIS OF LAMELLAR SYSTEMS: APPLICATIONS TO MYELINATED NERVE SHEATHS. R. Vargas and L. Mateu, Centro de Biofísica y Bioquímica, IVIC, Apdo. 21827, Caracas 1020-A, Venezuela, and V. Luzzati, Centre de Génétique Moléculaire, CNRS, 91198 Gif-sur-Yvette, France.

In order to undertake the study of order-disorder phenomena in myelinated nerve sheaths, a physical model has been proposed and an algorithm developed for the fast and accurate analysis of the whole diffraction pattern of lamellar systems [1].

The model consists of planar lamellae, all stacked into crystals with a variable repeat distance. Within each crystal the lamellae are parallel to each other. The following parameters are considered in the model: the average lattice repeat and its variance, the average number of unit cells per crystallite, the continuous intensity curve of a single motif the intensities of the diffuse scattering, and the fraction of lamellae that does not belong to the coherent diffraction domains.

The algorithm has been applied to a variety of X-ray scattering spectra from rat sciatic and optic nerve myelin [2,3]. Such data have been recorded and analyzed in a few minutes using a position sensitive detector and a personal computer. A precise determination of the structural parameters of myelin and as evolution as a function of the age of the rats has also been analyzed.

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P5.1 CRYSTAL STRUCTURE SOLUTION OF MG-LEUCITES FROM SYNCHROTRON X-RAY POWDER DIFFRACTION DATA. R. J. Cernik[t], A.M.T. Bell[t,2], P. E. Champness[3], A. N. Fitch[2,*], C.M.B. Henderson[3], S. C. Kohn[4], and S.A.T. Redfern[3].

A previously unknown low-symmetry leucite structure has been determined from synchrotron x-ray powder diffraction data collected on a hydrothermally synthesized leucite analogue of stoichiometry $K_2MgSi_5O_{12}$. Laboratory based x-ray powder diffraction and ^{29}Si nmr work showed that this structure is a distorted version of the known cubic (Ia3d) leucite structure and that all 12 tetrahedral (T) Mg and Si sites are ordered. Electron diffraction showed that the low-symmetry structure was monoclinic (P2_1/c). The cubic structure in the monoclinic cell was used as a starting model for Rietveld refinement of this structure. The refinement was able to determine on which two of the T sites Mg were situated from differences in the T-O bond lengths. The synchrotron x-ray diffraction data were able to distinguish quite clearly between the Mg and Si sites.

Rietveld refinement has also been carried out on a dry synthesized, cubic (Ia3d) $K_2MgSi_5O_{12}$ leucite analogue, in this structure the T sites are disordered.

[t]SERC Daresbury Laboratory, Warrington, WA4 4AD, UK.
[2]University of Keele UK.
[3]University of Manchester UK.
[4]University of Warwick UK.
[*]Now at ESRF, Grenoble, France.

P5.2   CRYSTAL STRUCTURE REFINEMENTS OF $M_3Al_2O_6$
(M = Ca, Sr, Ba) USING NEUTRON POWDER DIFFRACTION DATA
– AN EXAMPLE STUDY FROM THE NEW HB-4 DIFFRACTOMETER
AT THE HFIR, OAK RIDGE[*]. B. C. Chakoumakos, J. A. Fernandez-Baca,
and G. A Lager[1], Solid State Division, Oak Ridge National Laboratory, Oak
Ridge, TN 37831, U. S. A., [1]Department of Geology, University of Louisville,
Lousiville, KY 40292, U. S. A.

$Ca_3Al_2O_6$ is a major component of Portland cements, and the conversion of
the aluminate to the hydrogarnet structure is an important hydration reaction
in the setting of these cements. Detailed structural studies of isostructural
alkaline earth aluminates are underway to elucidate their reactivity and to
examine the systematics of Al-O bond length and Al-O-Al angle variations
among tetrahedral aluminates. Room temperature powder diffraction patterns
were collected for the title compounds in the form of dense ceramic rods using
a step size of 0.05° over the range of 11° to 135° 2Θ with a wavelength of
1.400 Å. These compounds are cubic, $Pa3$, Z = 24, and are described by
48 structural parameters using isotropic thermal vibration parameters. Their
crystal structure consists of isolated 6-membered tetrahedral aluminate rings
cross- linked by the alkaline earth ions. Rietveld structure refinements were
completed, employing a Pseudo-Voigt peak shape function, and a total of
12 profile and background parameters. For M = Ca, a = 15.2588 (4) Å,
V_{cell} = 3552.7 (2) Å^3, R_{exp} = 0.0496, R_{wp} = 0.0618, for 1444 reflections.
Comparison of the refined structural parameters with a previous single crystal
x-ray structure determination of the Ca analog (Mondal & Jeffery, 1975, Acta
Cryst. B 31, 689) show excellent agreement. For M = Sr, a = 15.8544 (4)
Å, V_{cell} = 3985.2 (2) Å^3, R_{exp} = 0.0294, R_{wp} = 0.0446, for 1614 reflections.
The present refinement of the Sr analog is essentially the same as an earlier
refinement employing the Voigt peak shape function (Chakoumakos et al.,
1992, Acta Cryst. C 48, in press). For M = Ba, a = 16.512 (2) Å, V_{cell} = 4502
(1) Å^3, R_{exp} = 0.0497, R_{wp} = 0.0955, for 1829 reflections. This refinement is
not as good as those for the Ca and Sr analogs. The assumption that the Ba
analog is isostructural (based on the similarity of the powder pattern) may
be incorrect, or the sample purity may not be as good. Both possibilities are
being explored.

P5.3   STRUCTURE DETERMINATION OF THE LOW- AND
HIGH-PRESSURE FORMS OF Mg_3BN_3 FROM X-RAY POWDER DATA.
H. Hiraguchi, H. Hashizume, S. Nakano[*] and O. Fukunaga[*], Research Lab-
oratory of Engineering Materials, Tokyo Institute of Technology, Nagatsuta,
Midori, Yokohama 227, Japan. [*]Fac. of Engineering, TIT.

The crystal structures of magnesium boron nitride Mg_3BN_3 in the low- and
high-pressure forms have been determined ab initio from X-ray powder data.
The strategy used is to decompose the powder pattern to estimate individ-
ual reflection intensities, calculate Patterson functions and build trial-and-
error structure models, which are refined by the Rietveld method. 54 and
21 reflections observed on a θ-θ diffractometer were used to solve the struc-
tures of $Mg_3BN_3(L)$ and $Mg_3BN_3(H)$ respectively. Low-pressure $Mg_3BN_3(L)$
has a hexagonal cell (space group $P6_3/mmc$, $Z=2$) with $a=3.54453(4)$ and
$c=16.0353(3)$ Å while the high-pressure $Mg_3BN_3(H)$ cell is orthorhombic
($Pmmm$, $Z=1$) with $a=3.0933(2)$, $b=3.1336(2)$ and $c=7.0005(5)$ Å. The two
structures commonly include linear N-B-N molecular anions, considered to
play a role in the catalytic properties of the material in the reaction convert-
ing hexagonal boron nitride into a cubic form.

A very similar structure has been obtained by applying standard direct
methods to the same $Mg_3BN_3(L)$ intensity data. High-quality electron density
maps have been calculated for this form using the maximum entropy method
and the phased structure factors. A separate application of the maximum
entropy program MICE [C. Gilmore et al.: Acta Cryst. A47, 830 (1991)]
to the unphased structure factors yielded a similar map, in support of the
determined structure of $Mg_3BN_3(L)$.

P5.4   STRUCTURAL CHARACTERIZATION OF WEBERITE-
RELATED ANTIMONATES $A_2Sb_2O_7$ (A = Pb,Sr,Ca,Cd). S. A. Ivanov,
Karpov'Institute of Physical Chemistry, 103064 Moscow K-64, Russia.

The discovery of ferroelectricity in weberite-related $Pb_2Sb_2O_7$ crystals
initiated a search for similar compounds with unusual dielectric properties.
The title compounds appear to be good candidates for application as
ferroelectrics. Investigation of the crystal chemistry of these antimonates
may be beneficial in developing a better understanding of the origin of
ferroelectricity. With this in mind, the features of the crystal structure of
$A_2Sb_2O_7$ complex oxides have been thoroughly studied by X-ray powder
diffraction. Diffraction patterns were collected with a URD-63 diffracto-
meter at room temperature between 12 and 132°2θ using CuK$_{\alpha}$-radiation
and analyzed with the Rietveld profile refinement technique (the DBW-3.2s
computer program). The structures of the $A_2Sb_2O_7$ samples are basically
similar: the Sb ions are octahedrally coordinated whereas the A2+ cations
are 8-coordinated and lie in square prisms and hexagonal bipyramids. The
existence of ferroelectric properties at room temperature only in $Pb_2Sb_2O_7$
can be related to the features of crystal structure. The important role played
by the effective size of the A2+ cations in determining the structural
parameters, and the electrostatic effect of the lone pair of electrons on
Pb2+ are also discussed.

A^{2+}	a, A	b, A	c, A	S.G.	<a>	R_h/R_{Sb}
Pb	7.484(1)	7.857(1)	10.426(2)	I2cm	5.351	1.93
Sr	7.455(1)	10.369(2)	7.684(1)	Imma	5.295	1.85
Ca	7.298(1)	10.213(2)	7.459(1)	Imma	5.179	1.68
Cd	7.302(1)	10.207(2)	7.249(1)	Imma	5.131	1.61

P5.5   CHARGE TRANSFER AND COMPRESSION EFFECTS IN DOPED
Y-123. S. Eriksson[†,#], C. Ström[†], P. Berastegui[†], L.-G. Johansson[†], A. Simon[#], Hj.
Mattausch[#], [†]Dept. of Inorganic Chem., S-412 96 Göteborg, SWEDEN, [#]MPI fur
Festkörperforschung, 7000 Stuttgart 80, GERMANY.

In most high-T_c superconductors the number of positive holes in the CuO_2 sheets to a
great extent determines the superconducting properties. By introducing dopant atoms or by
preparation of oxygen deficient samples drastic structural changes occur. The changes can
in many cases be correlated with a charge transfer in the structure and thus to a change in
T_c.

The POLARIS instrument at ISIS, RAL, England has been used for collection of high
quality TOF neutron powder diffraction data. In all cases solid state sintered single phase
samples of doped Y-123 have been analyzed by the Rietveld method. The structural
changes caused by the various dopant atoms have been correlated with the change in
properties (See table 1). Compression effects (Sr^{2+}) can be separated from charge transfer
effects (Co^{3+}, Al^{3+}, Fe^{3+}) and the two effects seem to be additive. Raman spectroscopic
investigations, susceptibility and Hall effect measurements confirm the picture obtained
from the analysis of the neutron data.

Table 1. Some characteristic distances in $Y_1Ba_{2-y}Sr_yCu_{3-x}Co_xO_{7-d}$. Cu1 is the chain
copper, Cu2 the plane copper, O2 and O3 the plane oxygens and O4 the apex oxygen.

x/y	(Cu2-O2,3)	(Ba-O4)$_z$	Cu1-O4	Cu2-O4	T_c
0.00/0.00	0.269	0.299	1.853(2)	2.291(5)	92
0.00/0.20	0.270	0.297	1.847(3)	2.282(3)	90
0.00/0.60	0.269	0.295	1.846(2)	2.250(3)	86
0.00/1.00	0.267	0.299	1.843(3)	2.224-(3)	81
0.05/0.00	0.259	0.304	1.854(5)	2.306(6)	89
0.15/0.00	0.228	0.343	1.836(5)	2.345(6)	76
0.30/0.00	0.216	0.349	1.838(6)	2.352(7)	30

Data will also be presented for the materials $YBa_{2-y}Sr_yCu_{3-x}Me_xO_{7-d}$ (Me= Co^{3+}, Al^{3+})
and $Y_{1-y}Ca_yBa_2Cu_{2.7}Co_{0.3}O_{7-d}$. The introduction of Ca^{2+} at the Y^{3+} site reintroduces
holes in the CuO_2 planes in Y-123 doped with cobalt. From the large number of samples
investigated it has been possible to obtain a firm understanding of the structure-property
relations in the Y-123 system.

P5.6   APPLICATION OF HIGH-RESOLUTION POWDER NEUTRON DIFFRACTION TO THE DETERMINATION OF THE NUCLEAR AND MAGNETIC STRUCTURES OF $(Y_{1-x}Ca_x)Ba_2Fe_3O_{8-\delta}$. Q. Huang, P. Karen, V. Karen, J.W. Lynn, A.D. Mighell, I. Natali-Sora, N. Rosov & A. Santoro. Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, U.S.A.

The nuclear and magnetic structures of $(Y_{1-x}Ca_x)Ba_2Fe_3O_{8-\delta}$ have been determined as function of temperature, calcium doping, and oxygen stoichiometry, using data collected with the high-resolution five-counter neutron diffractometer at the Reactor of the National Institute of Standards and Technology. The general atomic configuration in the unit cell of this compound is similar is that of the superconductor $YBa_2Cu_3O_7$. For $\delta = 0$ the symmetry of $(Y_{1-x}Ca_x)Ba_2Fe_3O_8$ is tetragonal and the iron ions corresponding to the Cu chains have octahedral coordination since all the oxygen sites on the basal plane are fully occupied. For $\delta > 0$, the oxygen vacancies occur on the basal plane and in such a way that some of the iron ions remain octahedrally coordinated and some assume a square-planar coordination similar to that of the chain copper ions in the 1,2,3 compound. This means that oxygen is eliminated preferentially only in one direction of the basal plane and, as a consequence, the symmetry of the compound becomes orthorhombic. The magnetic structure is based on a unit cell related to that of the nuclear structure by an axis transformation of matrix $(1\bar{1}0/110/002)$. The iron moments are coupled antiferromagnetically within each FeO_2 layer. The coupling along the c-axis, however, depends on the values of x and δ.

P5.7   A NEUTRON POWDER DIFFRACTION STUDY OF THE STRUCTURAL CHANGES IN $Tl_{2-x}Ba_2CuO_{6-d}$ AND $Tl_{2-x}Ba_2CaCuO_{8-d}$ RELATED TO THE OXYGEN AND THALLIUM CONTENT. C. Ström*, S. Eriksson*†, and L.-G. Johansson*, *Dept. of Inorganic Chem., S-412 96 Göteborg, SWEDEN.   †MPI für Festkörperforschung, 7000 Stuttgart 80, GERMANY.

The thallium-based high T_c superconductors with two Tl-O layers have tetragonal symmetry except for $Tl_2Ba_2CuO_{6-d}$ (Tl-2201), which exists in both tetragonal and orthorhombic form. Tl-2201 shows a remarkably wide range in T_c (0-90K) depending on preparative conditions. It is known that changes in T_c and cell parameters can be correlated with changes in oxygen content. $Tl_{2-x}Ba_2CaCuO_{8-d}$ (Tl-2212) shows no phase transition but it does show a large variation in the c axis length dependent on the method of preparation.

In this investigation we have studied the structural changes by neutron diffraction of samples with different thallium and oxygen content in order to simulate different synthetic conditions.

High quality neutron diffraction data were collected at the spallation source ISIS, RAL, UK, using the high flux, medium resolution powder diffractometer POLARIS. Data were collected from twelve solid state sintered single phase samples of $Tl_{2-x}Ba_2CuO_{6-d}$ and $Tl_{2-x}Ba_2CaCuO_{8-d}$ with different x and d, and analyzed by the Rietveld method.

Results from the analysis show that the tetragonal-orthorhombic phase transition in Tl-2201 is a function of both the oxygen and thallium content. The samples with high thallium deficiency are tetragonal. Orthorhombic samples with small thallium deficiency become tetragonal after oxygen treatment. The structural changes are largest in the Tl-O layers. Tl-2212 shows a shortening of the c-axis when the thallium and oxygen content increase. However, the changes in bondlengths are not additive. Structural and T_c data will be presented for Tl-2201 and Tl-2212.

K6.1 DEVELOPMENT OF HIGH PRESSURE POWDER DIFFRACTION ON SYNCHROTRON SOURCES. Y. Fujii, Institute of Materials Science, University of Tsukuba, Tsukuba, Ibaraki 305 and now Institute for Solid State Physics, The University of Tokyo, 7-22-1 Roppongi, Minato-ku, Tokyo 106, Japan.

By using pressure to directly control inter-atomic interactions, we have studied crystal structures newly stabilized at high pressures and related phase transition phenomena. At high pressures, hydrostaticity is no longer maintained because of the freezing of the pressure-transmitting fluid. This fact necessarily leads us to the use of powder specimens for diffraction experiments to obtain reliable intensity data. The relatively small aperture of a high-pressure apparatus seriously limits the accessible reciprocal space; therefore, powder diffraction has another advantage of allowing the collection of as much diffraction data as possible in such a restricted geometry. In order to enable us to obtain reliable intensity data from the powder specimen, the following systems dedicated to synchrotron radiation x-ray diffraction experiments at the Photon Factory have been developed cooperatively by the High-Pressure Group in Japan:

- (1) SR (BM, MPW on the 2.5 GeV Storage Ring, 10-20 keV)
  - Diamond-anvil cell (at low/high temperatures)
  - Imaging-plate, SSD (energy-dispersive mode)
  - Data acquisition software,

- (2) SR (BM on the 6 GeV Accumulation Ring, 10-100 keV)
  - Multi-anvil apparatus (high temperatures, MAX-80 & -90)
  - SSD (energy- or angle-dispersive mode)

The precise structure determination of molecular crystals and metals has enabled us to study precursory behavior, structural instability, and structural-similarity/scaling-rule uniquely driven by pressure.

O6.1 HIGH TEMPERATURE X-RAY DIFFRACTION DETERMINATION OF PHASE TRANSITIONS IN LARGE GRAIN ALLOYS. O. B. Cavin*, C.R. Hubbard, and P.J. Maziasz, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge TN 37831-6064, Phone (615) 574-5121.

HTXRD studies of the phase equilibria of alloys as a function of temperature depend. upon the ability to accurately follow the intensity of particular lines. Large grain size introduces the additional complication of spotty patterns due to poor particle statistics yielding ambiguous or uncertain intensities. To overcome this limitation, a rocking curve method has been used which involves sample oscillation about the goniometer axis. We have studied inherently large grained intermetallic aluminides, some of which have two ordered lattice types (DO_3 and B2) and have determined the transition temperatures of each type. These temperatures are required for modeling the alloys and for predicting ideal forming temperatures. By carefully choosing the alloying additions, the properties of these alloys can be controlled. Changes in the transition temperatures have been observed which makes them more attractive for specific applications.

O6.2 NEUTRON POWDER DIFFRACTION AT THE HIGH FLUX ISOTOPE REACTOR AT OAK RIDGE *. J. A. Fernandez-Baca[1], H. A. Mook[1], H. S. White[2], B. S. Hoffheins[3], and B. C. Chakoumakos[1], [1]Solid State Division, [2]Computing and Telecommunications Division, [3]Instrument and Control Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, U. S. A.

A new multidetector neutron powder diffractometer has been installed at the HB4 beam tube at the High Flux Isotope Reactor at Oak Ridge National Laboratory. The diffractometer design currently includes a 12' collimator in the primary reactor beam, a flat Ge (1 1 5) monochromator crystal and 32 equally spaced (2.7°) ^{3}He counters, with 6' Soller-slit collimators, that can be scanned to obtain a diffraction pattern between 11° and 135° 2θ. A new, vertically focusing, Ge monochromator will be installed in the near future. The monochromator angle is usually fixed at $2\theta_M = 80°$ to select neutrons of a wavelength of 1.4 Å. The instrumental resolution for this configuration has a minimum of $\Delta d/d = 2 \times 10^{-3}$. The instrument control is performed from a microVax computer, which is also used for data visualization and analysis. In order to allow for different users' preferences various Rietveld analysis codes are available. These include RIETAN (by F. Izumi), LHPM1 (by R. J. Hill and C. J. Howard) and GSAS (by A. C. Larson and R. B. Von Dreele). Various tests of the performance of the diffractometer have been completed successfully and this instrument is now available for routine use. These tests included the crystal structure refinements for a number of powder diffraction standards (Ni, Si, Al_2O_3, ZrO_2, etc). and a variety of oxides: $M_3Al_2O_6$, M = Ca, Sr, Ba; $Sr_3Al_2(D_4O_4)_3$; $MCuSi_4O_{10}$, M = Ca, Sr, Ba; and $PbAl_2Si_2O_8$. The best fits were obtained using the pseudo-Voigt peak shape function.

P6.1 A NEW FOURIER DIFFRACTOMETER AT THE IBR-2 PULSED REACTOR - HIGH RESOLUTION, HIGH INTENSITY, HIGH ACCURACY. A.M.Balagurov, Laboratory of Neutron Physics, JINR, 141980 Dubna, Moscow Reg., Russia.

Two neutron Fourier diffractometers are in practical operation at present: the mini-SFINKS facility[1] in Gatchina (Russia) and FSS[2] in Geesthacht (Germany). They use the Reverse Time Of Flight (RTOF) method[3] of data acquisition and have good prospects in structural and residual stress studies. It is anticipated that a new high resolution Fourier diffractometer (HRFD) will commence operation in Dubna (Russia) at the IBR-2 pulsed reactor in 1992[4]. At this diffractometer the RTOF mode will be also used, and precise structural studies of powders with very high resolution and high intensity are proposed as the main area of interest. The $\Delta d/d$ resolution of 0.0005 is provided by the high frequency of neutron beam modulation (~150 kHz); a high intensity is due to the high transmission of the Fourier chopper (~0.25) and the large solid angle (0.1 sr) of the time focused detector system. The additional modulation of the beam by neutron source pulses leads to a considerable (by 10 or more times) decrease in the correlation background level compared to a Fourier diffractometer at a steady state reactor. The high total neutron flux at the sample position enables studies of very small samples and time resolved experiments. The measurement time for a complete diffraction pattern is determined by the $\Delta d/d$ resolution and in the low-resolution limit (1%) is much less than one minute. The opportunities for structural studies with the new Fourier diffractometer are expected to be similar to those of the HRPD at the RAL and, in addition, the new instrument offers the possibility of optimizing the experimental conditions with respect to resolution and intensity.

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P6.2   THE HIGH RESOLUTION NEUTRON POWDER DIF-FRACTOMETER AT JRR-3M. Y. Morii, K. Fuchizaki(*), S. Funahashi, N. Minakawa, Y. Shimojyo and A. Ishida(**), Department of Physics, Japan Atomic Energy Research Institute, Tokai, Ibaraki 319-11, Japan

The characteristics of the High Resolution Neutron Powder Diffractometer (HRPD) installed and studied recently at the JRR-3M are reported. Some experiments performed with the HRPD are also presented. The HRPD is an ILL-D2B type diffractometer which has the following features. (1) a fine collimation system: 6'/12' - 20'/40' - 6', (2) pseudobent germanium 331 and copper 311 monochromators with take-off angles of 89° or 130°, (3) Sixty four 3rd collimator and detector systems placed at every 2.5° of diffraction angle. Typical diffraction patterns can be taken over scattering angles from 5° to 160° in a few hours, depending on the sample material, and analyzed by the Rietveld profile refinement method or others. For the case of a monochromator take-off angle of 130°, the angular resolution, FWHM, of the diffraction peaks is less than 0.35° over the whole scattering angle range. The best resolution is 0.2° at the focusing position, which corresponds to 0.2% in $\Delta d/d$. Various kinds of materials such as high T_c superconductors, ionic conductors, metals, carbon clusters etc. have been studied with the HRPD. Among them, $La_2NiO_{4.00}$ was investigated between 610K and 6K. The crystal structure below 60K was determined to be orthorhombic, and the magnetic moment of Ni^{2+} was evaluated to be about 1.5 μ_B at 80 K and 1.7 μ_B at 6K through a Rietveld analysis.
(*) present address: Department of Physics, Faculty of Science, Kyusyu University; Fukuoka 812 Japan
(**) present address: Nuclear Plant Department, NKK corporation; Yokohama 230 Japan

P6.3   THE HIGH RESOLUTION POWDER DIFFRACTOMETER AT THE NIST REACTOR. E. Prince, A. Santoro, I. G. Schröder & J. K. Stalick. Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, U. S. A.

A new, state-of-the-art, high-resolution, neutron powder diffractometer has recently been installed at the NIST research reactor. Because optimum powder patterns require differently shaped curves of resolution as a function of d spacing for crystals with different unit cell sizes, this instrument has provision for producing monochromatic beams with ~ 1.5Å wavelength at three different monochromator takeoff angles. A 75° takeoff angle is most useful for large unit cell crystals such as zeolites, where there is comparatively little information at short d spacings. On the other hand, a 120° takeoff angle is necessary for crystals with small, but low symmetry, unit cells. The instrument has 32 detectors spanning an angle of 155°. Focusing monochromators allow optimum use of reactor neutrons without significant degradation of peak shape. The instrument will be used for the determination and refinement of the structures of a wide range of technologically important materials.

P6.4   NEUTRON POWDER DIFFRACTION TO 20 GPa. J. S. Loveday, R. J. Nelmes, Department of Physics, University of Edinburgh, Edinburgh EH9 3JZ, UK, J. M. Besson, G. Hamel, Université Pierre et Marie Curie, 4 Place Jussieu, 75252 Paris, Cedex 05, France, and S. Hull, the ISIS Facility, Rutherford Appleton Laboratory, Chilton, Oxfordshire, UK

The maximum pressure accessible to neutron diffraction techniques has until recently remained in practice at the $\approx$ 3 GPa available with McWhan type alumina clamp cells. This has limited the study of a range of classic neutron problems (magnetism, structures containing light atoms, disorder, etc). To address this problem we have been developing an opposed-anvil cell, able to take samples of sufficient size for crystallographic studies to pressures in excess of 20 GPa. This cell is now in use on the POLARIS powder diffractometer at the UK spallation neutron source ISIS.

The cell consists of a compact 200t hydraulic ram applying pressure to a sample contained between two opposed anvils. These are made either of tungsten carbide or, for the highest pressures, sintered-diamond. A toroidal anvil profile is used and allows a sample of $\approx$ 100 mm³ volume to be taken to 10 GPa or 30 mm³ to be taken to 20 GPa.

This geometry has required the development of new shielding procedures to obtain the optimum signal to background and a means to correct for the complicated attenuation by the anvils. This work is still in progress, but the results of a number of preliminary structural studies will be presented, including the pressure dependence of the structure of D_2O ice VIII and the high-pressure phases of bismuth .

P6.5   HIGH-PRESSURE STRUCTURAL STUDIES USING ANGLE-DISPERSIVE TECHNIQUES. M. I. McMahon, R. J. Nelmes, P. D. Hatton, R. O. Piltz, J. Crain and D. R. Allan, Department of Physics, The University of Edinburgh, Edinburgh, EH9 3JZ, UK.

During the past year, an image-plate (IP) system has been developed at SRS Daresbury with the objective of performing full Rietveld refinement of high-pressure crystal structures using angle-dispersive powder-diffraction techniques. Considerable care has been taken to reduce background scatter and eliminate contaminant diffraction lines, and this has enabled us to collect data of unrivalled quality. In our initial studies of the III-V semiconductor InSb, we have discovered two new high-pressure phases, and have shown that the long-accepted structures of two more high-pressure phases are incorrect. Furthermore, the very high sensitivity of the IP system, coupled with anomalous dispersion techniques, has enabled us to determine whether the high-pressure phases of InSb are ordered or not - a question that has remained unanswered for 30 years. The latest results on InSb will be described, along with new results from another III-V material, GaSb. Very recently, we have managed to obtain high-quality diffraction patterns from the low-Z semiconductor B_4C. The effect of pressure on this interesting material will also be presented.

P6.6   AUTORIET: SOFTWARE FOR "AUTOMATING" RIETVELD ANALYSIS OF TIME-OF-FLIGHT NEUTRON POWDER DIFFRACTION DATA. F. J. Rotella, Intense Pulsed Neutron Source (IPNS) Division, and R. L. Hitterman, Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439, U. S. A.

In certain types of experiments (e.g., time-resolved diffraction measurements), the rate at which the data are collected far outstrips the speed with which the said data can be analyzed by standard (user-intervention) Rietveld techniques. In an effort to deal with this situation, computer software has been written to automate Rietveld analysis at IPNS by optimizing data analysis time, much in the same way that computer control of instruments has optimized data collection times and sample throughput. AUTORIET is similar to software which has been used for the automated analysis of time-resolved neutron powder data from the Institut Laue-Langevin for the past eight or so years. It is a VAX/VMS DCL command file that, with auxiliary FORTRAN programs, creates the appropriate files that will be used for input to the IPNS Rietveld analysis software, controls the execution of the software, and creates summaries of the analysis for all sets of data considered. The user analyzes the initial data set via standard Rietveld techniques; AUTORIET will analyze the remaining sets in a user-specified order, based on the assumption that the starting parameters for any given data set are the results from the analysis of the preceding set. The summaries that are produced may be collated into files from which the variation of any parameters with respect to one or more other parameters in the model may be obtained in tabular form and/or graphically using visualization software on the IPNS VAX Cluster or on a workstation or personal computer. Although AUTORIET was designed to be used specifically with software in the IPNS Rietveld analysis package, adapting it for use with other Rietveld analysis software in a VAX/VMS environment should be fairly easy. The use of AUTORIET in the analysis of in situ diffraction data from a working electrochemical cell and in the determination of thermal expansion coefficients of bismuth-based superconductors will be presented.

P6.7   HIGH-PRESSURE X-RAY DIFFRACTION STUDY OF INDIUM UP TO 93 GPA   K. Takemura, *National Institute for Research in Inorganic Materials, Tsukuba, Ibaraki 305 JAPAN*

Structural change of indium under pressure has been precisely studied up to 93 GPa at room temperature using a diamond-anvil cell, synchrotron radiation and an imaging plate. The c/a axial ratio of the face-centered tetragonal (fct) structure increases with pressure, reaching a maximum around 24 GPa ($(c/a)_{max} = 1.091$) and decreases with further compression [1]. At around 45 GPa, indium undergoes a structural phase transition. The powder diffraction pattern of the high-pressure phase can be indexed to a face-centered orthorhombic cell with the lattice constants of a=3.771, b=3.841, c=4.142 Å at 93 GPa. By assuming plastic deformation of indium under uniaxial stress, the observed diffraction intensity can be successfully explained, including the asymmetric peak profiles. The model assumes a successive slip of the (111) plane in the $[01\bar{1}]$ direction, which often occurs in the fcc metals subjected to uniaxial stress. The estimated volume change at the fct-fco transition is close to zero, offering a possibility that the transition is of the second order. The transition was reversible with respect to pressure.

The lowering of the lattice symmetry of indium under pressure can be understood on the basis of the universal phase diagram for the group IIIb elements calculated by Hafner and Heine with the pseudopotential method [2].

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P6.8   HIGH RESOLUTION SYNCHROTRON X-RAY POWDER DIFFRACTION WITH A LINEAR POSITION-SENSITIVE DETECTOR. D. E. Cox and G. C. Smith, Brookhaven National Laboratory, Upton, N.Y. 11973, U.S.A., L. W. Finger, Carnegie Institution, Washington, D. C. 20015, U.S.A., and A. P. Jephcoat, Oxford University, Oxford OX13PR, England.

Some synchrotron x-ray powder diffraction experiments are described in which a multiwire proportional detector was operated in the "escape-peak" mode with a 90% Kr- 10% CO_2 gas mixture at 4 bar. Under these conditions, it is possible to achieve intrinsic position resolution of about 30 µm for 17.4 keV X-rays, compared to about 150 µm for the "photo-peak" mode [1]. A gating signal can be applied to the position-sensing electronics to provide energy discrimination. The detector, which has an active length of 10 cm, was mounted vertically on the scattering arm of the diffractometer at beam-line X7A at the NSLS about 45 cm from the sample axis. With well-crystallized reference materials such as CeO_2 and Al_2O_3 and an incident 0.7Å x-ray beam with horizontal and vertical dimensions of 4.0 and 0.2 mm respectively, the angular resolution obtained in the center of the detector was about 0.03° FWHM, although there was considerable degradation at the ends because of parallax effects. High quality data suitable for precision lattice parameter determination, auto-indexing or Rietveld analysis can be obtained in as little as a few minutes by stepping the detector at 2° intervals and combining the central segments into a composite data set. Some applications will be described, including diffraction experiments in diamond - anvil cells.

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P6.9   TIME RESOLVED POWDER DIFFRACTION.   H. Fuess and P. U. Pennartz*, TH Darmstadt FB21/Strukturforschung. *now: ENRAF/NONIUS-Solingen Germany.

Complete powder diffraction patterns were recorded at the synchrotron source DORIS II (HASYLAB, Hamburg) in time intervals of milliseconds. Recording of the diffracted intensity was achieved by a commercial diode-array detector (EG & G-PARC) in Debye-Scherrer geometry. A time resolution of 200 msec for a snapshot of 40° (2θ) was obtained by a toroidal mirror and a conventional (n,-n) silicon (111) double crystal monochromator.

The detector consists of 1024 single silicon (p/n) diodes each 25 µm wide and 2 mm high. The detecting window is therefore only 25 mm long. In order to record a large region of the diffraction pattern the detector is mounted very close to the sample. The angular resolution of this setup is comparable with that of a standard x-ray powder pattern from a conventional source, but the time resolution is about 10 times higher than with conventional systems. Enlarging the detector to sample distance will increase the angular resolution and decrease the integral time resolution.

A multilayer monochromator (OVONICS, 200/W/Si double layers of 2.05 nm thickness each) decreased the measurement time for a whole pattern to 2.5 msec. The multilayer system, however, increased the energy-bandpass by two orders of magnitude. Thus the angular resolution is about 10 times worse than the angular resolution of conventional powder diffractometers (Pennartz et al., J. Appl. Cryst, 1992, in press).

In contrast to previous work (Wong et al. 1990, Science, **249**, 1404) we aimed at the recording of a complete powder pattern and not just a small region or a single Bragg-reflection. Therefore we tested two other detectors (Pennartz et al. Z. Kristallogr. 1989, **186**, 227-230), including the blade-anode ENRAF-NONIUS/INEL 120° curved CPS120. The performance of these detectors has been tested using conventional sealed x-ray tube sources and rotating anodes, and also at the synchrotron.

P6.10 A NEW METHOD FOR POWDER DIFFRACTION
STUDIES. Mark A. Beno and Gordon S. Knapp,
Material Science Division. Argonne National Laboratory,
Argonne Il 60439, U.S.A.

Powder diffractometers are among the most widely used of all analytical
instruments. Seemann-Bohlin diffractometers, the most widely used type,
measure data serially, one angle at a time with many angles scanned to
observe the entire diffraction pattern. Linear and area detectors lack energy
resolution and so can suffer from high backgrounds particularly if materials
containing elements which fluoresce are to be studied. In addition, the one
dimensional detectors developed to date do not possess sufficient resolution
for highly accurate structural studies particularly in the case of synchrotron
based x-ray powder diffraction instruments.
The method we employ uses a focusing diffracted beam monochromator in
combination with a multichannel detector which allows us to reduce the
background and to take data in parallel, providing a large improvement over
conventional diffractometers. In
our technique the incident x-rays
(from a laboratory source or
synchrotron) fall on a flat plate or
capillary sample and are intercepted
by a bent focusing monochromator
which has the focus of the bend
at the sample surface. The beam
emerging from the bent crystal
monochromator falls on a linear or
2-dimensional detector. Initial tests
show that this method can be used
to acquire the data much faster and
with much higher sensitivity.
Existing instruments can be easily
modified to employ this method.

Results from laboratory and synchrotron sources will be presented for
crystalline and glassy materials along with schemes for rapid data collection
employing other monochromator and detector configurations.
Work at Argonne National laboratory is sponsored by the US Department of
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under contract W-31-109-ENG-38.
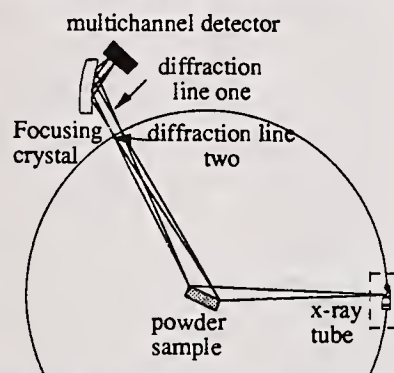
P6.11 HIGH TEMPERATURE NEUTRON POWDER DIF-
FRACTION FURNACES, S. L. Town, R. L. Davis* & E. H. Kisi[†],
ANSTO, PMB 1, Menai, NSW 2234, Australia, *AINSE, PMB 1, Menai,
NSW 2234, Australia, †Division of Science & Technology, Griffith Uni-
versity, Nathan, Queensland 4111, Australia.

A neutron powder diffraction furnace, capable of temperatures to 1100°C
has been tested and commissioned for use on the medium and high reso-
lution powder diffractometers at ANSTO. The design of this is discussed,
and data from Y_2BaCuO_5 and CaF_2 are presented; problems in refining
thermal parameters are highlighted. A new furnace, capable of temper-
atures to 1750°C is currently being developed, and the design is detailed
here.

Tariq Ahmedali
McGill University
3450 University St.
Geol. Sciences
Montreal, H3A 2A7
Canada

Charles Albert
SCM Chemicals
3901 Fort Armistead
Baltimore, MD 21226
U.S.A.

David Allan
Univ. of Edinburgh
Mayfield Rd.
Edinburgh EH9 3JZ
UK

J. Paul Attfield
Univ. of Cambridge
Dept. of Chemistry
Lensfield Rd.
Cambridge CB2 1EW
UK

Neil A. Ball
University of Manitoba
Dept. of Geological Sci.
Rm 24, Wallace Bldg.
Winnipeg, Manitoba R3T 2N2
Canada

Davor Balzar
NIST
325 Broadway 853
Boulder, CO 80303
U.S.A.

Maurizio Bellotto
CISE-Tecnologie Innovative
Via R. Emilia, 39 - Segrate
(MI) PO Box 12081
Milan, 20134
Italy

Mark Beno
Argonne National Lab.
9700 S. Cass Ave.
Bldg. 223, A225
Argonne, IL 60439
U.S.A.

Jean Francois Bérar
Ecole Centrale Paris
voie des Vines
92295 Châtenay
France

J.G.M. van Berkum
Laboratory of Materials Sci.
Rotterdamseweg 137
2628 AL Delft
The Netherlands

Giovanni Berti
Univ. Pisa
Via S. Maria 53
I-56100 Pisa
Italy

David L. Bish
Los Alamos National Lab.
Mail Stop D469
Los Alamos, NM 87545
U.S.A.

Julie A. Borchers
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Alun Bowen
Defence Research Agency
Materials & Structures Dept.
RAE Farnborough
Hants, GU14 6TD
UK

Hans Boysen
Inst. für Kristallographie
Universität München
Theresienstr. 41
8000 München 2
Germany

Paul Brand
University of Maryland/NIST
Bldg. 235, Rm. E151
Gaithersburg, MD 20899
U.S.A.

Bob Broach
UOP
50 East Algonquin Rd.
Des Plaines, IL 60017
U.S.A.

Xianhui Bu
Chemistry Department
Univ. of California (SB)
Santa Barbara, CA 93106
U.S.A.

O. B. Cavin
Oak Ridge National Lab.
PO Box 2008
Bldg 4515, MS 6064
Oak Ridge, TN 37831
U.S.A.

Bryan Chakoumakos
Oak Ridge National Lab.
PO Box 2008
Solid State Division
Oak Ridge, TN 37831
U.S.A.

Lawrence Chatfield
Alfred Univeristy
Inst. for Ceramic Super-
   conductivty BMH-1-55
Alfred, NY 14802
U.S.A.

Robert Cheary
University of Technology
Broadway
Sydney, 2007
Australia

Yakov E. Cherner
Institute of Physics
194 Stachki Ave.
344104 Rostov-on-Don
Russia

Feng Chen
Rider College
2083 Lawrenceville Rd.
Chemistry Dept.
Lawrenceville, NJ 08648
U.S.A.

Rosaria Chiappetta
University of Calabria
Arcavacata Di Rende
87030 Cosenza
Italy

Steve Chipera
Los Alamos National Lab.
Mail Stop D469
Los Alamos, NM 87545
U.S.A.

Chang Sun Choi
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Jim Cline
NIST
Materials Bldg., Rm A256
Gaithersburg, MD 20899
U.S.A.

Alan A. Coelho
University of Technology
51 Stanley St. East
Sydney 2010 NSW
Australia

Palola Comodi
Piazza Universita
Dipartimento di Scienze della
  Terra
I-06100 Perugia
Italy

David E. Cox
Brookhaven National Lab.
Physics 510B
Upton, NY 11973
U.S.A.

M. K. Crawford
DuPont Co.
Experimental Station
E356/209
Wilmington, DE 19889-0356
U.S.A.

Yongshan Dai
American Museum of Natural
  History (Mineral Sciences)
Central Park West at 79th St.
New York, NY 10024-5192
U.S.A.

Bill David
Rutherford Appleton Lab.
Chilton, Didcot, Oxon
OX11 0QX
UK

Briant L. Davis
X-Ray Diffraction Laboratory
South Dakota School of
  Mines and Technology
Rapid City, SD 57701
U.S.A.

W. Depmeier
University of Kiel
Mineralogisch-Petrogr. Inst.
Olshausenstr 40
D-W-2300 Kiel
Germany

Janet Douglas
Freer Gallery of Art
12th & Independence Ave.
Washington, DC 20560
U.S.A.

Lars Eriksson
Stockholm University
Arrhenius Laboratory
S-106 91 Stockholm
Sweden

Sten Eriksson
University of Göteborg
Dept. of Inorganic Chem.,
Sven Hultius gata
S-41296 Göteborg
Sweden

Nils O. Ersson
Uppsala University, Inorg.
  Chemistry, Box 531
S-75121 Uppsala
Sweden

Michael Estermann
ETH Zürich, Inst. of Cryst.
ETH-Zentrum NOF
CH-8092 Zürich
Switzerland

Howard T. Evans
U.S. Geological Survey
National Center 95Q
Reston, VA 22092
U.S.A.

Melanie E. Feather
Smithsonian Institution
CAL-MSC
Washington, DC 20560
U.S.A.

Jaime Fernandez-Baca
Oak Ridge National Lab.
PO Box 2008 MS 6393
Oak Ridge, TN 37831-6393
U.S.A.

Paul Fewster
Philips Research
Cross Oak Lane
Redhill, RH1 5HA
UK

Larry Finger
Geophysical Lab.
5251 Broad Branch Rd., NW
Washington, DC 20015
U.S.A.

C. M. Foris
Dupont Central Res. & Develp.
Experimental Station
Bldg. 228
PO Box 80228
Wilmington, DE 19880
U.S.A.

Joseph Formica
Siemens Analytical X-Ray
6300 Enterprise Lane
Madison, WI 53719-1173
U.S.A.

Mario Fornoff
International Centre for
  Diffraction Data
1601 Park Lane
Swarthmore, PA 19081
U.S.A.

Yasuhiko Fujii
University of Tokyo
Institute for Solid State
  Physics
7-22-1 Roppongi Minato-Ku
Tokyo 106
Japan

Klaus Futterer
Technische Univ. of Berlin
Inst. für Mineral. & Krist.
Ernst-Reuter-Platz 1
D-1000 Berlin 12
Germany

Bill Giessen
Northeastern University
Boston, MA 02115
U.S.A.

Ganesh Golikeri
Unilever Research
45 River Rd.
Edgewater, NJ 07020
U.S.A.

Donald Goodwin
Charles Supper Co.
25 Tech Circle
Natick, MA 01760
U.S.A.

Berton Greenberg
Philips Laboratories
345 Scarborough Rd.
Briarcliff Manor, NY 10510
U.S.A.

Boualem Hammouda
NIST
Bldg 235,Rm E151
Gaithersburg, MD 20899
U.S.A.

George Harlow
American Museum of Natural
  History
Dept. of Mineral Sciences
Central Park West at 79th St.
New York, NY 10024-5192
U.S.A.

Richard Harlow
DuPont Co.
CRP E228/316D
Wilmington, DE 19880
U.S.A.

Hiroo Hashizume
Tokyo Institute of Technology
Research Lab. of Eng. Matls.
Nagatsuta, Midori
Yokohama 227
Japan

Daniel Häusermann
European Synchrotron
  Radiation Facility
B.P. 220
F-38043 Grenoble Cedex
France

Tomas Havlik
Technical University
Letna 9/A
Dept. Non Ferrous
CS-04385 Košice
Czechoslovakia

Peter Heaney
Princeton University
Guyot Hall
Princeton, NJ 08544
U.S.A.

Helmut Hermann
Inst. of Solid State Res.
Helmholtzstr. 20
Postfach 6
D-0-8027 Dresden
Germany

Alan Hewat
I.L.L.
156 X Grenoble 38042,
France

Rafael Hidalgo
Northeastern University
Boston, MA 02115
U.S.A.

Roderick Hill
CSIRO Mineral Products
PO Box 124
Port Melbourne
Victoria 3207
Australia

T. M. Holden
Chalk River Nuclear Lab.
Atomic Energy of Canada
Chalk River, Ontario K0J 1J0
Canada

Mark Holomany
International Centre for
  Diffraction Data
1601 Park Lane
Swarthmore, PA 19081
U.S.A.

Joe Hriljac
Brookhaven National Lab.
Dept. of Physics
Bldg. 510B
Upton, NY 11973
U.S.A.

Jingzhu Hu
Geophysical Laboratory
5251 Broad Branch Rd., NW
Washington, DC 20015-1305
U.S.A.

Qingzmen Huang
University of Maryland/NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

T. C. Huang
Alamaden Research K34/802
IBM Research Division
650 Harry Rd.
San Jose, CA 95120
U.S.A.

Camden Hubbard
Oak Ridge National Lab.
PO Box 2008
Bldg. 4515, MS 6064
Oak Ridge, TN 37831
U.S.A.

Diane Hues
NIST
Bldg. 222, Rm 1113
Gaithersburg, MD 20899
U.S.A.

Matti Järvinen
Lappeenranta Univ. of
  Technology
PO Box 20
SF-53851 Lappeenranta
Finland

William Jackson
Shell Development Co.
3737 Bellaire Blvd.
Room 210
Houston, TX 77025
U.S.A.

Ron Jenkins
International Centre for
  Diffraction Data
1601 Park Ave.
Swarthmore, PA 19081
U.S.A.

Quintin Johnson
Materials Data Inc.
PO Box 791
Livermore, CA 94551
U.S.A.

Arthur Jurgensen
Westinghouse
Savannah River Lab.
Aiken, SC 29808
U.S.A.

James Kaduk
Amoco Corporation
PO Box 3011 F-9
Naperville, IL 60566
U.S.A.

Walter Kalceff
University of Technology
PO Box 123 Broadway
Sydney NSW 2007
Australia

Vicky L. Karen
NIST
Bldg. 223, Rm A215
Gaithersburg, MD 20899
U.S.A.

Richard M. Kirchner
Manhattan College
Chemistry Dept.
Bronx, NY 10471
U.S.A.

Peter Klimanek
Mining Academy Freiberg/Sa.
PO Box 47
DO-9200 Freiberg/Sa,
Germany

Vladimir Kogan
Rostov-on-Don State Univ.
Zorge St. 5,
344044 Rostov-on-Don
Russia

Susan Krueger
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

George A. Lager
University of Louisville
Department of Geology
Louisville, KY 40292
U.S.A.

Vratislav Langer
Chalmers Univ. of Technology
Dept. of Inorg. Chemistry
S-41296 Göteborg
Sweden

J. Ian Langford
University of Birmingham
School of Physics & Space
  Research
Birmingham B15 2TT
UK

Armel Le Bail
Laboratoire des Fluorures
Université du Maine
72017 Le Mans Cedex
France

Kurt Leinenweber
SUNY at Stony Brook
Earth & Space Science
Stony Brook, NY 11794-2100
U.S.A.

Kenneth Liao
National Steel Corp.
1745 Fritz Dr.
Trenton, MI 48183
U.S.A.

Ulrich Loechner
Forchenweg 1
7100 Heilbronn
Germany

Daniel Louër
Université de Rennes
Laboratorie de Cristallochimie
Avenue de Général Leclerc
35042 Rennes Cedex
France

John Loveday
Rutherford Appleton
Bldg. R3
Chilton, Oxon OX11 0QX
UK

Charlotte Lowe-Ma
Naval Air Warfare Ctr-WPNS
Code 3851
China Lake, CA 93555
U.S.A.

Luca Lutterotti
Univerisitá di Trento
Dept. of Materials Engineering
Via Mesiano, 77
Trento, 38050
Italy

Jeff Lynn
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Paul Maistrelli
Dipartimento Di Fisica
Povo
Trento, 38050
Italy

Massimo Marezio
AT&T Bell Labs/CNRS
IC-211
Murray Hill, NJ 07974
U.S.A.

Curtis Martin
NSWC
10901 New Hampshire Ave.
Code R31
Silver Spring, MD 20874
U.S.A.

Lynne McCusker
Inst. of Crystallography
ETH-Zentrum
CH-8092 Zürich
Switzerland

Malcolm McMahon
The Univ. of Edinburgh
Dept. of Physics
Edinburgh EH9 3JZ,
UK

Jan van Mechelen
Koninklijke/Shell Lab
Badhuisweg 3 AG-Dept.
1031 CM Amsterdam
The Netherlands

Ron Medrud
Chevron Research &
  Technology Co.
100 Chevron Way
50-1254
Richmond, CA 94802
U.S.A.

Apurva Mehta
Princeton University
113 Guyot Hall
Princeton, NJ 08544
U.S.A.

Alan Mighell
NIST
Bldg. 223, Rm A215
Gaithersburg, MD 20899
U.S.A.

Evan Morgan
Babcock and Wilcox
PO Box 11165
Lynchburg, VA 24506
U.S.A.

Yukio Morii
Japan Atomic Energy
  Research Inst.
Tokai, Ibaraki, 319-11
Japan

Arndt Mücklich
Research Centre Rossendorf
P.O.B. 19
D(O)-8023 Dresden
Germany

Michael S. Nakhmanson
Comphys Lab Enterpise
NPO Bourevestnik
68 Mapookhtinsky Ave.
195272 St. Petersburg
Russia

Isabella Natali-Sora
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Fang-Ling Needham
International Centre for
  Diffraction Data
1601 Park Lane
Swarthmore, PA 19081
U.S.A.

Diane Nelson
Air Products and Chemical
7201 Hamilton Blvd
Allentown, PA 18195-1501
U.S.A.

Robert Newman
Dow Chemical
1897 G Bldg.
Midland, MI 48667
U.S.A.

John M. Newsam
Biosym Technologies Inc.
9685 Scranton Rd.
San Diego, CA 92121
U.S.A.

Jackie Nicol
University of Maryland/NIST
Bldg 235, E151
Gaithersburg, MD 20899
U.S.A.

Heinrich Oettel
Tech Univ. Freiberg
DO-9200 Zeunerstr 5
Freiberg/SA,
Germany

Rosemary Pacalo
SUNY at Stony Brook
Earth & Space Sciences
Stony Brook, NY 11794-2100
U.S.A.

John Parise
SUNY at Stony Brook
Earth & Space Sciences
Stony Brook, NY 11794
U.S.A.

Damodara Poojary
Texas A&M University
College Station
Dept. of Chemistry
College Station, TX 77840
U.S.A.

Ben Post
International Centre for
  Diffraction Data
1601 Park Lane
Swarthmore, PA 19081
U.S.A.

Jeffrey Post
Smithsonian Institution
10th & Constitution Ave., NW
NHB 119
Washington, DC 20560
U.S.A.

Henry Prask
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Charles Prewitt
Geophysical Lab.
5251 Broad Branch Rd., NW
Washington, DC 20015
U.S.A.

Edward Prince
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Angeline Rehfeldt-Oskierski
Siemens
7500 Karlsruhe 21
Germany

James Rice
Sun Co. RM/R&D
PO Box 1135
Marcus Hook, PA 19061
U.S.A.

Lee Robertson
Oak Ridge National Lab.
Bldg. 4500, MS 6118
Oak Ridge, TN 37831
U.S.A.

Stephen B. Robie
Scintag, Inc.
707 Kifer Rd.
Sunnyvale, CA 94086
U.S.A.

Nick Rosov
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Daphne Ross
Smithsonian Institution
10th & Constitution Ave.,NW
E408/NHB 119
Washington, DC 20560
U.S.A.

Frank J. Rotella
Argonne National Laboratory
9700 S. Cass Ave.
IPNS Division - Bldg. 360
Argonne, IL 60439
U.S.A.

J. Michael Rowe
NIST
Bldg. 235/A106
Gaithersburg, MD 20899
U.S.A.

John J. Rush
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Makoto Sakata
Nagoya University
Chikusa-Ku, Furo-cho 1
Nagoya, T464-01
Japan

Antonio Santoro
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

David Schiferl
Los Alamos National Lab.
MS C345
Los Alamos, NM 87545
U.S.A.

Julius Schneider
Inst. für Kristallographie
Universität München
Theresienstr. 41
D-8000 München 2
Germany

Lyle Schwartz
Materials Bldg./B309
NIST
Gaithersburg, MD 20899
U.S.A.

Julia Sigalovsky
Massachusetts Institute of
  Technology
77 Massachusetts Ave.
Rm 12-022
Cambridge, MA 02139
U.S.A.

Husinsyah Sitepu
Indonesian Institute of Tech.
JL Raya Puspiptek
Serpong Tangerang
Indonesia

Ewa Skrzypczak
Univeristy of Toledo
2801 W. Bancroft St.
Chemistry Dept
Toledo, OH 43606-3390
U.S.A.

Deane Smith
Penn State University
239 Deike Bldg.
University Park, PA 16802
U.S.A.

Lubomir Smrčok
Institute of Inorganic
  Chemistry
Slovak Academy of Sciences
Bratislava
Czechoslovakia

Robert Snyder
NYS College of Ceramics
Alfred University
Alfred, NY 14802
U.S.A.

Judith Stalick
NIST
Bldg. 235, Rm E151
Gaithersburg, MD 20899
U.S.A.

Eric Steel
NIST
Bldg. 222, Rm 1113
Gaithersburg, MD 20899
U.S.A.

Carin Ström
University of Göteborg
Dept. of Inorganic Chemistry
S-41296 Göteborg
Sweden

Mark Sutton
McGill University
3600 University St.
Montreal H3A 2T8
Canada

J. Timmers
Philips Research
Bedrokvem B/V Bldg. WY
2.22
PO Box 80.000
5600 JA Eindhoven
The Netherlands

Brian Toby
Air Products
7201 Hamilton Blvd.
RSD1 R1102
Allentown, PA 18195
U.S.A.

Hideo H. Toraya
Nagoya Institute of
  Technology
Asahigaoka
Tajimi 507
Japan

Sue Town
ANSTO PMB 1
Menai
NSW 2234
Australia

Judy Triphahn
UOP
50 East Algonquin Rd.
Des Plaines, IL 60017
U.S.A.

Shirley Turner
NIST
Bldg. 222, Rm 1113
Gaithersburg, MD 20899
U.S.A.

Václav Valvoda
Charles Univ., Prague
Ke Karlovu 5
121 16 Prague 2
Czechoslovakia

Rodolfo Vargas
I.V.I.C.
Apartado 21827
Caracas 1020 A
Venezuela

Mary Vickers
BP
Chertsey Rd.
Sunbury-on-Thames
TW16 7LN
UK

Jan W. Visser
Henry Dunantlaan 81
2614 GL Delft
The Netherlands

Robert Von Dreele
Los Alamos National Lab.
Lansce, MS H805
Los Alamos, NM 87545
U.S.A.

Wilem L. Vos
Geophysical Laboratory
5251 Broad Branch Rd., NW
Washington, DC 20015-1305
U.S.A.

Sue-Lein Wang
National Tsing Hua Univ.
Dept. of Chemistry
30043 Hsinchu, Taiwan
Republic of China

Donald Weidner
SUNY at Stony Brook
Earth & Space Sciences
Stony Brook, NY 11794-2100
U.S.A.

P.-E. Werner
Arrhenius Lab.
Stockholm University
S-10691 Stockholm
Sweden

Marianne Westdahl
Stockholm University
Arrhenius Lab.
S-10691 Stockholm
Sweden

Angus Wilkinson
University of California
Materials Dept.
Santa Barbara, CA 93106
U.S.A.

George Will
University Bonn
Poppelsdorfer Schloss
5300 Bonn
Germany

Eric Windsor
NIST
Bldg. 222, Rm 1113
Gaithersburg, MD 20899
U.S.A.

Winnie Wong-Ng
NIST
Materials Bldg., Rm A215
Gaithersburg, MD 20899
U.S.A.

Nicholas Wright
Edinburgh University
Edinburgh
Scotland

Takamitsu Yamanaka
Osaka University
College of Gen Educ.
1-1 Machikaneyama
Toyonaka Osaka, 560
Japan

R. A. Young
Georgia Inst. of Tech.
School of Physics
Atlanta, GA 30332
U.S.A.

Marek Zakrzewski
Procter & Gamble
Pharmaceuticals 13815-0191
Norwich, NY 13815
U.S.A.

Pier Franceseo Zanazzi
Piazza Universita
Dipartimento di Scienze
  della Terra
I-06100 Perugia
Italy

Lev Zevin
Ben-Gurion University
Institute for Applied Research
PO Box 1025
Beer-Sheva
Israel

Xinyu Zhang
Northeastern University
Boston, MA 02115
U.S.A.

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U.S. DEPARTMENT OF COMMERCE
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JCPDS-International Centre for Diffraction Data, 1601 Park Lane, Swarthmore, PA 19081, USA
International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, UK
National Institute of Standards and Technology, Gaithersburg, MD 20899, USA

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The proceedings of the international conference Accuracy in Powder Diffraction II present the invited papers and abstracts of the papers contributed to the conference, which was held at NIST, Gaithersburg, Maryland, during May 26-29, 1992. The conference was organized by the Commission on Powder Diffraction of the International Union of Crystallography, and was jointly sponsored by NIST, JCPDS—International Centre for Diffraction Data and the International Union of Crystallography. The proceedings contain 25 invited papers and 73 contributed abstracts. The program of the conference was divided into six topics: Phase Identification and Quantification; Accuracy and Standards; New Developments in Software and Data Analysis; Profile Fitting, Decomposition and Microstructural Effects; Novel Applications and Structural Science; and New Developments in Hardware, Including Detectors, and Studies under Non-ambient and Time-resolved Conditions. A ceremonial session was devoted to a tribute to the late William Parrish and his contributions to powder diffraction. In addition, there were two tutorial workshops organized by the JCPDS—International Centre for Diffraction Data, one on diffractometer sensitivity and one on automatic indexing methods.

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