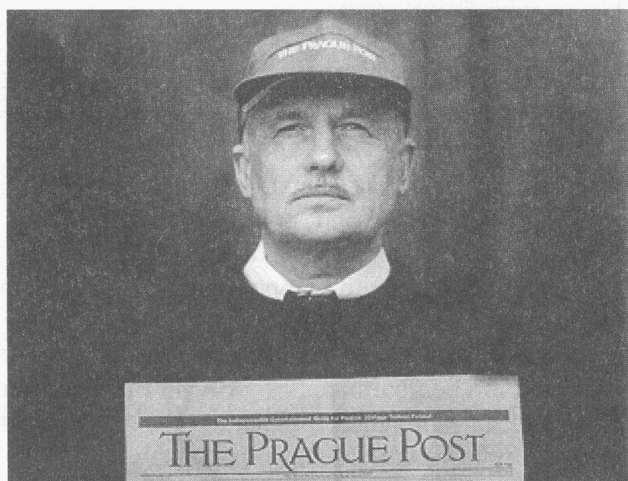




COMMISSION ON POWDER DIFFRACTION INTERNATIONAL UNION OF CRYSTALLOGRAPHY

NEWSLETTER No. 16, JUNE 1996

TEN POSTCARDS FROM PRAGUE



We are sending you these postcards of selected results of our work to inform you about our activities in the field of X-ray powder diffraction. As you will see, the range of current problems solved in our laboratory is broad, which stems a natural advantage of powder diffraction to be applicable in many different ways for many useful ends. If something among these points is interesting to you, don't hesitate to contact us, please. You can also visit our WWW pages at <http://rtg-1.karlov.mff.cuni.cz/kfpo/osa/eosa.htm>.

The group of X-ray powder diffraction people at the Faculty of Mathematics and Physics, Charles University in Prague, Bohemia, a small country that contrary to Shakespeare's belief [W. Shakespeare, **The Winter's tale** (1610-1611), Act III Scene 3] does not lie on the sea shore.

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1. Magnetic Multilayers

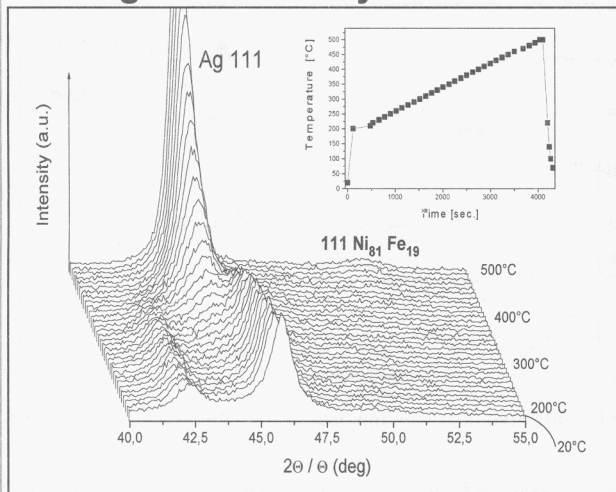


Figure 1: *In-situ* annealing of $[\text{Ag}(27\text{\AA})/\text{Ni}_{81}\text{Fe}_{19}(7\text{\AA})]_{10}$ multilayer and changes in XRD profiles at the high-angle range.

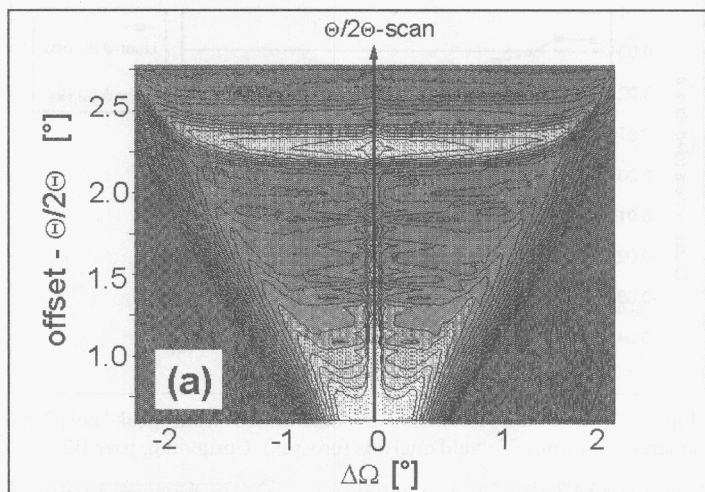


Figure 2: Low angle XRD area-scan of a NiCo/Au multilayer. Maxima in diffuse scattering refer to correlated roughness.

The study of synthetically grown multilayer structures has now received increased interest as a result of novel optical, electrical and magnetic properties observed in these systems. We have studied structure of magnetic multilayers in the systems Ag/ $\text{Ni}_{81}\text{Fe}_{19}$, Cu/Co, Au/Ni, Au/ $\text{Ni}_x\text{Co}_{1-x}$ and Au/Fe. The information from the low angle region ($0 < \theta < 8^\circ$), where the method of X-ray reflectivity is used, and from the high angle region ($\theta > 8^\circ$), where methods of powder diffraction are applicable, have been combined. During the annealing process shown in Fig. 1 the multilayer is broken down and separate peaks of Ag and NiFe are visible. In Fig. 2 a map of diffuse scattering is presented. We perform quantitative refinement of roughness, vertical and lateral correlation lengths (reflectivity, Ω and offset scans) using the DWBA approach extended by Holy for layered systems [1]. If possible we recommend the combined measurement in both mentioned regions because the extracted information is partially complementary.

2. Thin Films

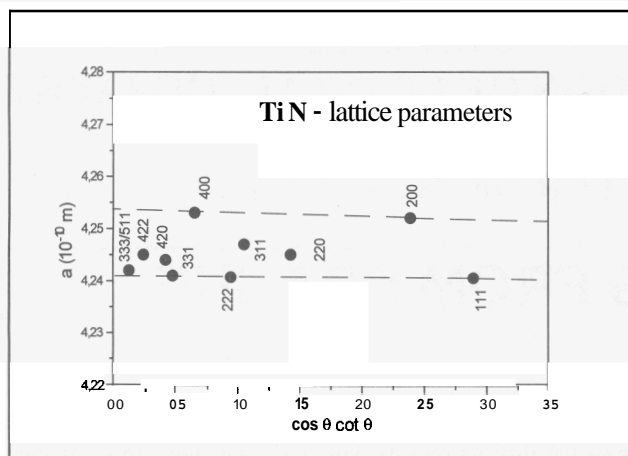


Figure 3. Cohen-Wagner plot of lattice parameters of cubic TiN calculated from individual reflections.

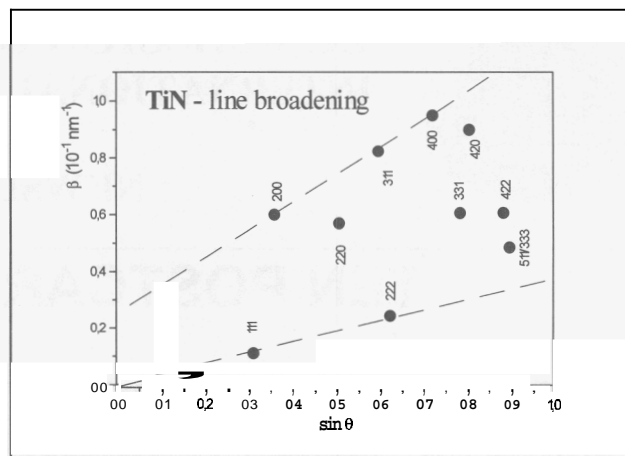


Figure 4. Williamson-Hall plot of line broadening for the same sample of TiN.

These two figures illustrate how even a cubic material deposited (in this case by magnetron sputtering) as a thin film can behave like a material with lower symmetry. The values of lattice spacing and strain/size broadening in grains oriented with their (200) planes parallel to the substrate are systematically larger in comparison with the values found in (111) grains. Such behaviour was found in porous films, whereas the films with compact microstructure behave in exactly the opposite way [2]. This crystallographic anisotropy of microstructural properties in inhomogeneous polycrystalline thin films is not completely understood till now. The existence of one principal direction of deposition of atoms, thermodynamically unstable deposition conditions, permanent ion bombardment of growing films and generally large residual stresses conserved in the films lead to the creation of an inhomogeneous lattice deformation which is dependent on crystallographic grain orientation with respect to the sample surface.

3. Intensity and Rietveld Round Robin

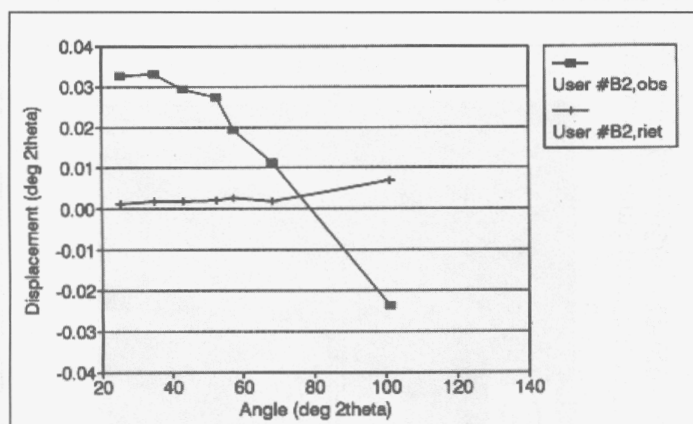


Figure 5. Peak displacements as determined from peak positions (squares) and from Rietveld analysis (crosses). Corundum, user B2.

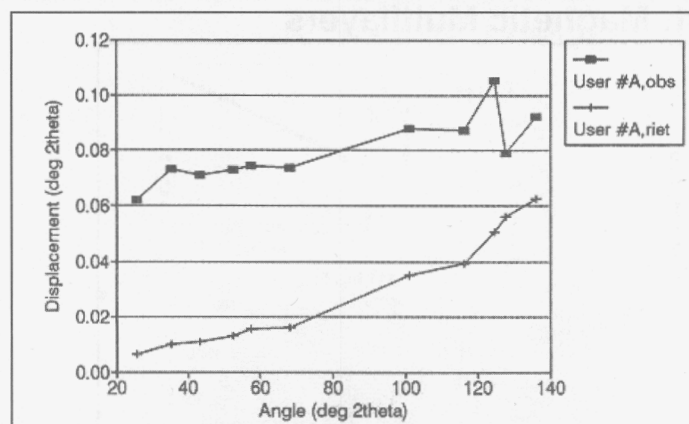


Figure 6: Results obtained from the data of user A.

The results show that the Rietveld whole pattern fitting program can correct for diffractometer misalignment, like in the case of user B2 (Fig.5). However, in some cases, like in the case of user A, these corrections fail. The residual angle-dependent peak displacements are clearly seen in Fig.6 even in the data obtained by the Rietveld fitting. Such incomplete corrections were found in cases when the sample displacement was too big or when the quality of the data was poor from the point of view of the Rietveld program. Three Round Robin tests were organized in 27 laboratories in close co-operation with Ron Jenkins from the ICDD [3]. The aim of these tests was to show the sensitivity of powder diffractometers, the influence of instrumental parameters and the method of data collection and reduction. Sintered and powder samples of corundum were used in these tests to show also the influence of sample preparation. Usage of a certified powder diffraction standard is strongly recommended to improve accuracy of measurements.

4. Preferred grain orientation - texture

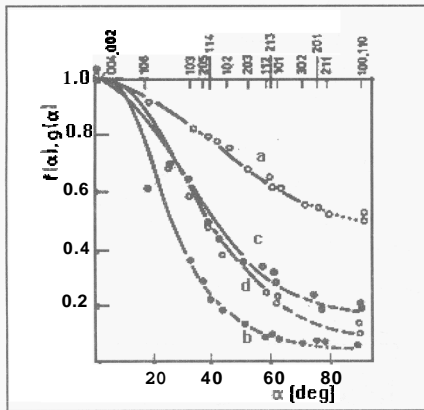


Figure 7. Orientation distribution of (001) planes of Mg. Lines - texture goniometer data, circles - calculated values from (hkl) reflections.

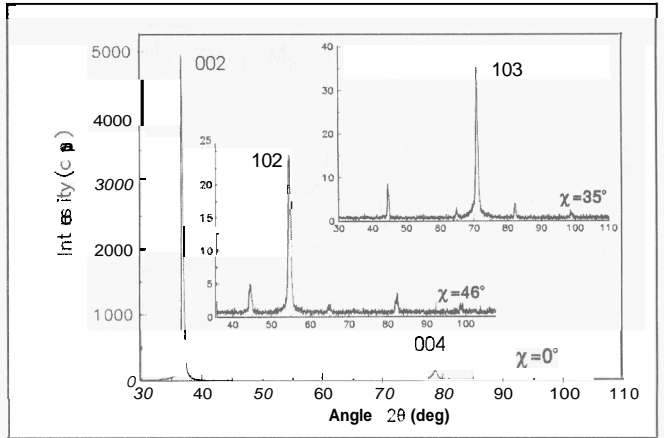


Figure 8. Joint texture refinement: XRD patterns of a strongly textured thin film of Zn measured at three sample inclinations $\chi = 0, 35$ and 46°

The effect of texture may strongly influence accurate intensity measurements. The experiments [4] with powder samples of magnesium lead us to the following empirical formula capable describing the observed orientation distribution function: $\exp[-G(1-\cos^n\alpha)]$. Such simple empirical functions are especially useful in the case of strongly textured samples with a low number of free parameters (in contrast to the more flexible expansions in spherical harmonics). Thin films ordinarily belong to this class of materials, as can be seen in Fig.8. To enhance the intensity of reflections from planes which don't belong to the family of preferentially oriented planes measurements at several sample inclinations should be done. Structure refinement based on the whole set of reflections measured at all sample settings is called Joint Texture Refinement. The application of simple empirical functions in calculations of texture corrected intensities in the POWLS program needed an extension of this code by numerical integration [5].

5. Alignment in magnetic field

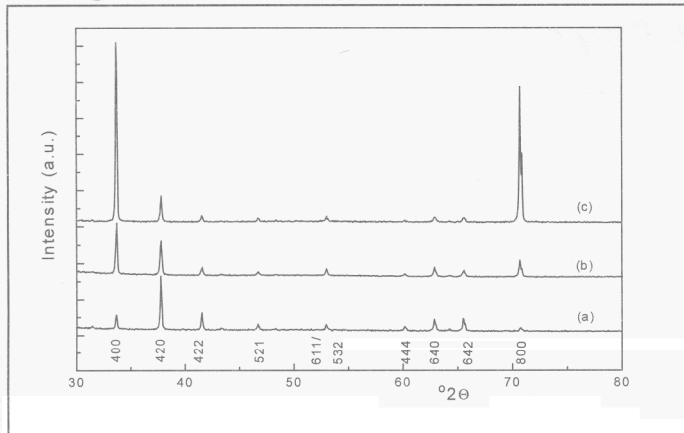


Figure 9: Changes in diffraction patterns of YIG indicate different degree of texture: non-oriented (a), oriented by magnetic impulses (b) and oriented in a steady magnetic field (c).

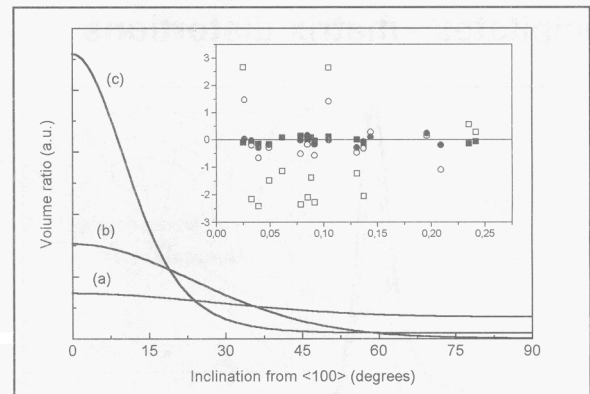
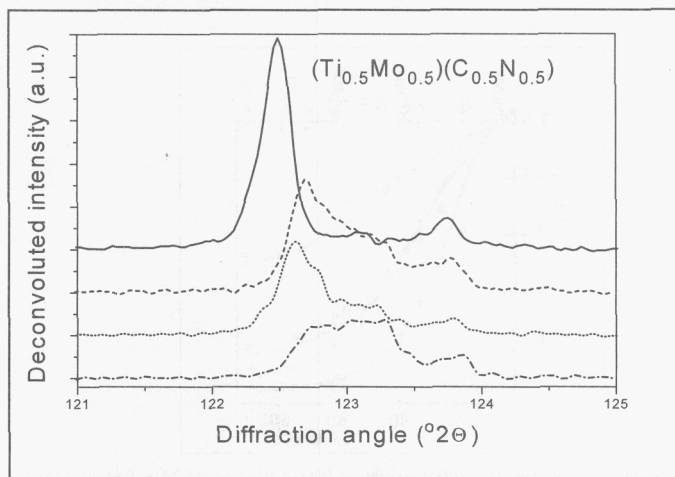
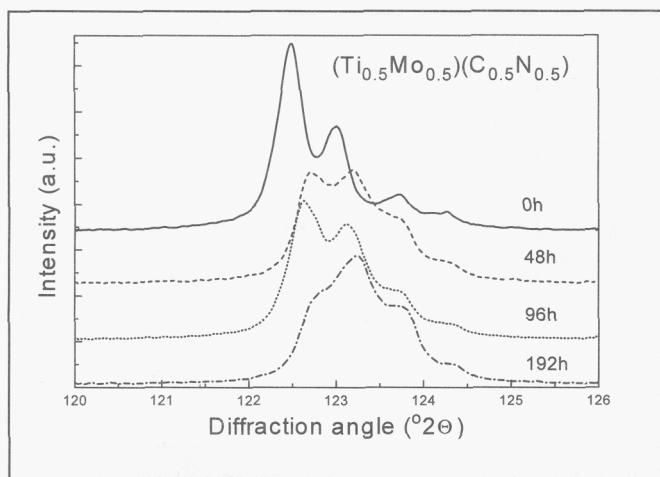


Figure 10: Orientation distribution of (100) planes in samples with different degrees of preferred orientation. The Wilson plot for sample C before and after texture correction is shown in the inset.

The efficiency of methods for aligning randomly oriented crystallites in a magnetic field [6] was tested. As a first approach, the orientation of crystallites of the cubic yttrium iron garnet (YIG, $\text{Y}_3\text{Fe}_5\text{O}_{12}$) was investigated. A sample that was oriented exclusively by the intrinsic magnetic anisotropy of YIG without applying an external magnetic field was used as a reference. That allowed us to separate the intrinsic magnetic anisotropy and the anisotropic response to the external magnetic field. The observed changes in diffracted intensities (Fig. 9) confirmed the desirable texture $\{100\}$. After the sample was oriented by magnetic impulses, the half-width of the Gaussian distribution of crystallites around the direction (100) was about 30° (Fig.10). A substantially higher degree of ordering with a half-width of about 12° was reached if the sample was rotated in a steady magnetic field. In the calculation of the preferred orientation, the empirical texture function according to [4] was applied taking into account partial multiplicity factors [7].

6. Microscopic homogeneity and composition of quarternary composites



Figures 11 and 12: Successive homogenisation of a hot-pressed mixture of (Ti,Mo)C and Ti(C,N) having the nominal composition of $(\text{Ti}_{0.5}\text{Mo}_{0.5})(\text{C}_{0.5}\text{N}_{0.5})$ after annealing at 1450°C for 2, 4 and 8 days.

The fcc quarternary compounds $(\text{Ti},\text{X})(\text{C},\text{N})$ containing molybdenum or tungsten atoms at the alternative metal sites are studied for their good mechanical properties and chemical stability. In our laboratory we investigate the microscopic homogeneity in these materials by means of XRD. These composites tend to create mixtures of the nitrogen-poor titanium molybdenum (tungsten) carbide and the titanium carbonitride, which do not have the same quality as the quarternary compounds. In materials that show a certain degree of decomposition we can determine the distribution in concentration of the particular components in individual grains. The EPMA with WDS or EDS that is commonly used for such a purpose cannot be applied, because the size of crystallites is less than $1\mu\text{m}$. To separate the unwanted instrumental line broadening, shown in Fig. 12, a deconvolution procedure based on the least-squares method was developed. The deconvoluted intensities are smoothed by modified Golay-Savitzky method.

7. Precipitates - matrix distortions

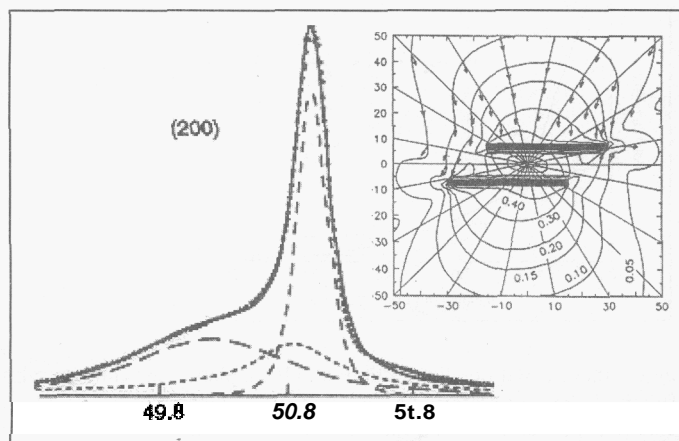


Figure 13: XRD profile measured on aged Cu-Be alloys fitted with a sum of Bragg peak, static diffuse scattering and quasiline. Inset: the calculated displacement field for precipitate stair step.

Aging in Cu-Be alloys leads to the growth of precipitates and consequently to large matrix distortions giving special diffraction effects. The problem is treated by partitioning of the complex profile into Bragg peak and two components of diffuse scattering according to Krivoglaz and Houska theory. Precipitate size and shape is determined by comparing the fitted profiles with the results obtained from a simplified elastic model including also precipitate stair-steps.

8. Clay minerals

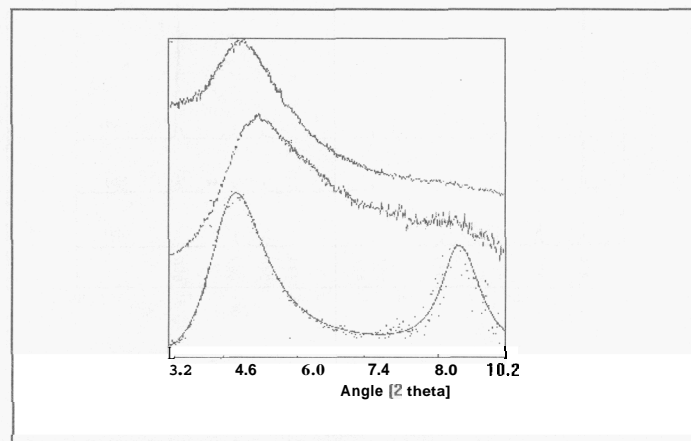


Figure 14: XRD profiles 001 and 002 of Al-13 pillared montmorillonite: measured (top), corrected for Lp-factor (middle), corrected for Lp-factor and square of structure factor (bottom).

XRD profile analysis of natural and pillared clay minerals has been developed for the case of low angle region of $2\theta < 10^\circ$ and broad 00/ reflections. The method, based on assuming the Lorentz-polarization and structure factor correction as a continuous function of diffraction angle, has shown a strong dependence of the peak position and peak shape on the line broadening and on the values of the structure factor in the given 2θ range.

9. High-T_c Superconductors

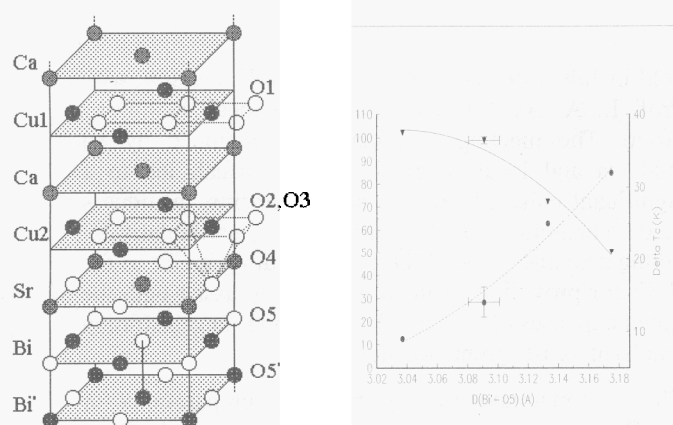


Figure 15: Critical temperatures (solid line) and width of the superconducting transition (dotted line) as a function of the distance D(Bi' - O5) of atomic planes (o - oxygen atoms, ● - heavy atoms).

Phase composition, structure parameters, changes in atomic configuration and in Cu oxidation state of high-T_c phase (Bi_{1-x}Pb_x)₂Sr₂Ca₂Cu₃O_y (2223 phase) were studied. Structural and crystallochemical analysis was based on the Rietveld structure refinement of XRD patterns. We found that the different superconducting qualities of the slowly cooled and quenched samples, both with dominant content of high-T_c phase 2223, can be related to the changes of atomic plane distances (see Fig. 15, left).

10. Ambient aerosol

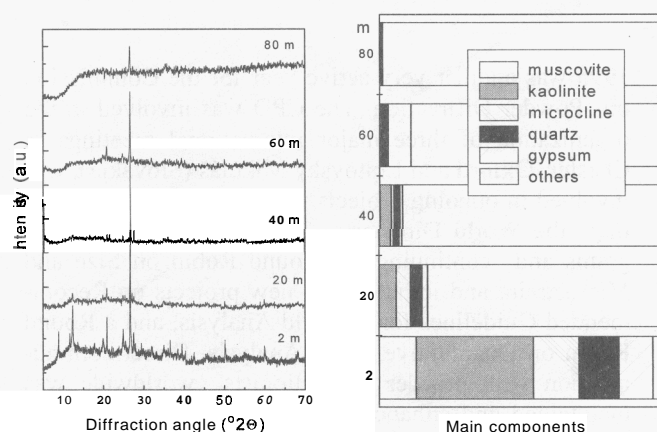


Figure 16: Relative amounts of minerals in ambient aerosol as a function of height above earth (others = amorphous particles, graphite, mullite, etc.)

The solid fraction of ambient aerosol collected at the different heights above the earth's surface was investigated by x-ray diffraction analysis. XRD enables the determination of the crystalline and amorphous particles of the fly ash (SiO₂, C, etc.) which are insoluble in the body liquids. Above 40 m, the light waste products of the coal burning power plants are significant and their effect on air and soil pollution can be specified.



From left: V. Valvoda, D. Janeba, P. Capkova, H. Sichova, L. Dobiasova, R. Kuzel, D. Rafaja

References

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- [7] P.Capkova, R.Peschar and H. Schenk, **J.Appl.Cryst.** 26(1993)449.

CHAIRMAN'S MESSAGE

1995 was another very active year for the Commission on Powder Diffraction. The CPD was involved in the organization of three major international meetings in Chester, Oxford and Liptovsky Mikulas (Slovakia), was involved in ongoing projects for maintaining and updating the World Directory of Powder Diffraction Programs and continuing the Round Robin on Size and Microstrain, and initiated two new projects on Recommended Guidelines for Rietveld Analysis, and a Round Robin on Quantitative Phase Analysis. Direct communication with powder diffractionists worldwide was maintained and enhanced with the production of two more issues of the CPD Newsletter and its wide dissemination via an increased mailing list and its installation on the World Wide Web.

Meetings - Workshops - Schools

After undertaking largely independent activities over the past six years, the CPD and the European powder diffraction community (in the form of the EPDIC series of meetings) started what is hoped to be an increasing level of cooperation. This interaction began with the invitation to the CPD Chairman, R. J. Hill, along with other members of the CPD, J. Fiala and D. Louer, to serve on the Programme Committee of the EPDIC-IV meeting in Chester in July 1995. The collaboration will continue with similar CPD involvement in the organization of EPDIC-V in Italy in 1997.

The CPD lent its support to the very successful International Meeting on Structure Solution from Powder Diffraction Data, held in Oxford in July, 1995 and organized by W. I. F. David and colleagues from the Rutherford Laboratory, U. K. CPD members L. B. McCusker, D. Louer and R. J. Hill presented invited lectures and workshops, and the IUCr provided a generous grant to the Organizing Committee. This conference was a very timely meeting with a very productive mixture of lectures and workshops that captured much of the excitement of this rapidly evolving area of powder diffraction.

The CPD was also involved in the International Conference on X-ray Powder Diffraction Analysis of Size/Strain, Macrostress and Texture held in Slovakia in August, 1995 under the local guidance of Dr. P. Sutta. CPD members J. Fiala, J. I. Langford and D. Louer and Chairman R. J. Hill served on the Program Committee, J. Fiala as Secretary, and the IUCr again provided support in the form of a grant.

CPD consultant and immediate Past Chairman, R. A. Young continued the series of CPD-organized three-day Rietveld Summer Schools with co-lectures R. B. Von Dreele and A.K. Cheetham. The latest in this series was

held in July 1995 at the Moscow State University, with Prof. L. A. Aslanov as head of the local organizing group. The meeting was very well attended by 86 students and local scientists and, as usual, led to a significant enhancement of the participants, awareness of the capabilities of Rietveld analysis, particularly by young scientists. The CPD is once again grateful to the IUCr for providing financial support to assist with the travel expenses of the main lecturers and for registration and living-costs grants for students.

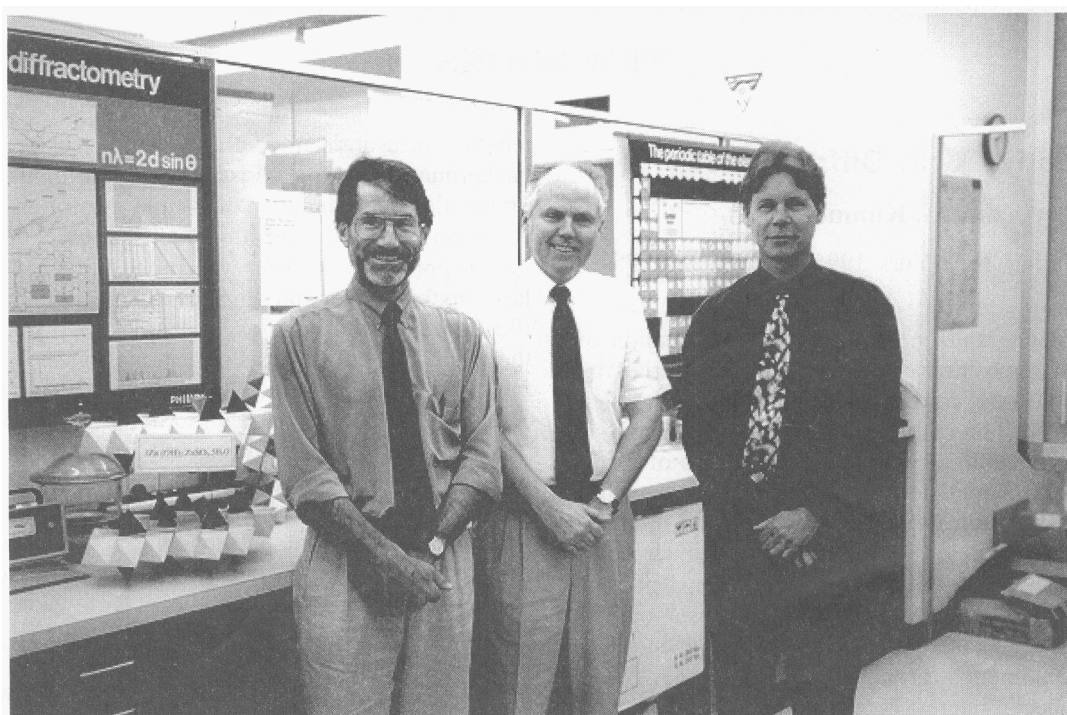
The CPD maintained a high profile in preparations for the IUCr Congress in Seattle through the involvement of its Chairman, R. J. Hill, and member D. E. Cox on the Programme Committee and with associated responsibility for the coordination of lecturers and other contributions to most of the powder diffraction related Microsymposia. The CPD's nominee, D. Louer, was invited by the IUCr to present a Plenary Lecture entitled Powder Diffraction - An Update, at the Congress, and the CPD-initiated Microsymposium entitled Materials VIII-Powder Diffraction was also accepted.

In a novel initiative, the CPD has combined forces with the 45th Annual Denver X-ray Conference to jointly organize the Powder Diffraction Satellite Meeting in Denver, August 3-8, 1996, associated with the XVII Congress in Seattle. Two members of the CPD, D. E. Cox and D. K. Smith, served on the Organizing Committee, and several CPD members and Consultants were involved as invited speakers or Workshop and special session organizers.

Projects

In conjunction with the Slovakia meeting, CPD member J. Fiala and R. L. Snyder (ICDD) co-chair a Task Group working on a Round Robin on Crystallite Size and Microstrain Determination. The preliminary results of studies on a widely circulated standard sample of a ceramic material containing inclusions of crystallites of known size were reported during the Slovakia meeting.

Continued organizational and financial support was provided by the CPD for the maintenance and updating of the World Directory of Powder Diffraction Programs, coorganized by S. Gorter (Netherlands) and D. K. Smith. This compilation has now appeared in two updated versions and includes summary descriptions and contact information for several hundred programs. It has become a major resource that is accessible to the world-wide powder diffraction community. The grant from the CPD has been used to enable the database to be even further expanded in scope and size, and to provide hardware for it to be distributed by anonymous FTP.



CPD Chairman Rod Hill, CPD Secretary Daniel Louer and Ian Madsen (L to R), pictured in the David Wadsley X-ray Laboratory at CSIRO Minerals Division in Melbourne, Australia

Two new projects were initiated during the year. One involves the production of summary guidelines and protocols for the collection and analysis of powder data for Rietveld analysis, and related to this, the identification of a new Rietveld-method standard material with higher complexity than the monoclinic zirconia sample used as a benchmark for the recently-completed CPD Rietveld Refinement Round Robin project. This work is being coordinated by CPD members L. B. McCusker and D. E. Cox. The second new project is being coordinated by D. K. Smith and R. J. Hill, and involves a new round robin survey of the accuracy, precision, capabilities and limitations of Quantitative Phase Analysis by diffraction methods. The selection of appropriate multiphase samples and discussion of the protocols for the survey are well advanced, with the recommendations planned for open discussion during a workshop at the Denver Powder Diffraction Satellite Meeting in August, 1996.

Newsletter

A novel feature of the CPD Newsletter availability now and in the future is its recent installation on the World Wide Web with access via a pointer from the IUCr Home Page. CPD member R. J. Cernik (Daresbury Laboratory, UK) has installed a copy of Newsletter 15 along with contact details and a brief introduction to the CPD and has agreed to continue this

service for subsequent issues. It is expected that this innovation will

ultimately provide even more widespread access and contributions to the activities of the CPD during the next Triennium. The CPD is grateful to the Daresbury Laboratory for its agreement to support the distribution of the Newsletter in both hard and soft form in this manner.

R.J.Hill. CPD Chairman

Meeting of the CPD Chairman and Secretary

The Chairman and Secretary of the CPD took advantage of a rare opportunity to discuss CPD issues in person during Daniel Louer's visit to Australia in early February. Dr Louer was in Sydney to give an invited lecture and workshop on powder diffraction during a meeting of the Australian X-ray Analytical Association. He followed this meeting with an extended visit to the CSIRO Division of Minerals in Melbourne, at the invitation of Ian Madsen of CSIRO, where he presented an excellent workshop on pattern indexing and participated in wide-ranging discussions on powder diffractometry.

This was a very valuable visit since it also enabled Daniel Louer and Rod Hill to talk about progress with the CPD's Round Robin on Quantitative Phase Analy-

NEW BOOKS

"Quantitative X-ray Diffractometry"

by L. S. Zevin and G. Kimmel

Springer Verlag, New York 1995, ISBN 0-387-94541-5, 372 pp.

This is a monograph on quantitative determination of the phase composition of polycrystalline materials by X-ray diffraction. It consists of six chapters. In the first chapter, the position of X-ray diffraction phase analysis among other techniques for the determination of phase composition is elucidated and the historical development of quantitative X-ray powder diffraction (QXRD) phase analysis is described. The second chapter deals with the physical background of X-ray powder diffraction in general with special emphasis on the QXRD. The third chapter is devoted to instrumentation: various experimental arrangements are reviewed in detail and special interest is paid to the Bragg-Brentano diffractometer. The most valuable and at the same time the most voluminous parts of the book are the fourth and the fifth chapters which address the main methodological and practical aspects of the QXRD phase analysis. Internal standard method, external standard method, standardless methods, doping method, dilution method, calibration techniques, diffusion of errors, reference intensity ratios, implementation of calculated powder diffraction patterns, overlapping peaks, full diffraction pattern approach, combination of X-ray diffraction and chemical data, determining crystallinity of polymers and analysis of low mass samples are discussed in detail in chapter number four, while more practical aspects of the QXRD phase analysis like in-

tensity measurement, definition and subtraction of background, counting statistics, detection limit, sample preparation, determination of sample absorption, pattern decomposition and simulation, real structure effects and their suppression are dealt with in the fifth chapter. The last, sixth chapter presents a number of examples illustrating various (mainly industrial) applications of the QXRD phase analysis of ceramics, glass ceramics, minerals, ashes, cement, metals and alloys, thin films and coatings, aerosols and airborne dusts and pharmaceuticals. The book addresses all aspects of QXRD phase analysis in great detail. The depth of the elaboration as well as the width of the scope (423 references) and the mastery of the presentation are unparalleled in the world scientific literature. The work reassumes a previous work of the first author - L. S. Zevin, L. L. Zavyalova "Quantitative X-ray Phase Analysis" (in Russian), Nedra, Moscow 1974 - that was the first and many years the only monograph devoted solely to QXRD phase analysis in the world. The Zevin-and-Kimmel's book "Quantitative X-ray Diffractometry" will be an indispensable everyday working manual for all laboratories doing QXRD phase analyses on a professional level and one of the fundamental reference books of the world's crystallographic literature. It is written very carefully so that it will serve well in teaching X-ray crystallography, too, promoting in this way QXRD phase analysis which is beyond any doubt one of the most valuable contributions of crystallography to metallurgy, mineralogy, analytical chemistry and materials technology.

J. Fiala

MEETING REPORTS

4th Regional Czecho-Slovak Conference on Powder Diffraction

Liptovsky Mikulas, Slovakia,
20 - 22 September 1995

The conference organized by the Department of Inorganic Chemistry of the Faculty of Chemical Technology of Slovak Technical University in Bratislava, Institute of the Inorganic Chemistry of the Slovak Academy of Sciences, Military Academy Liptovsky Mikulas and Czech and Slovak Crystallographic Association with participation of powder diffraction crystallographers from Slovakia and Czech Republic was held at the Department of Physics of the Military Academy in Liptovsky Mikulas, Slovakia, in September 20-22, 1995. The main aim of this conference was to serve as a communication pool for practitioners in powder diffraction through lectures, demonstrations, discussion ses-

sions and posters. Abstracts from all these lectures and posters were published in a booklet (22 pp) given to each participant.

After the initial lecture presenting curriculum vitae of W. C. Röntgen - discoverer of X-rays, the program of the conference continued with interesting contributions on the influence of incoherent dispersion on the diffraction pattern (N. Ganey), line profile analysis on thin films (P. Sutta et al.), X-ray diffraction study of the decay products of tetragonal martensite (Q. Jackuliak), in situ powder diffraction study of chemical reactions (L. Benes), a weighting scheme in Rietveld refinement (L. Smrcek), texture development in non-oriented electrotechnical silicon-containing steels (M. Cernik), testing free-spread software from WWW-server : general structure analysis software (A. Dvorsky), lattice parameters in zeolite of faujasite type (V. Jorik), X-ray diffraction study of nanostructured materials (V. Kavcansky et al.), information content of the

powder diffraction pattern and how it depends on data acquisition conditions (A. Buchal), determination of the magnetic moment orientation by X-ray diffraction (L. Caplovic) and computer control of an X-ray diffractometer (J. Haluska).

The program of the conference was supplemented by a visit to the Department of Physics of the host Academy, where the X-ray laboratory aroused interest of the participants.

J. Stankovic'

13th International Conference on X-ray Diffraction Analysis of Raw Materials.

Belgorod, Russia, 17-20 October 1995

The conference was dedicated to the memory of the late Prof. V. A. Frank-Kamenetsky, whose name was inseparably linked with the conferences of raw materials. He was one of the founders of the Commission on X-ray Diffraction of Raw Materials of the All-Union Mineralogical Society of the Russian Academy of Sciences and presided over all of the twelve past conferences of this series. In fact, he served as the Chairman of the Organizing Committee of the 13th Conference, too, but unfortunately died before the beginning of the conference.

A distinguishing feature of the present conference was that it took place in the year when the worldwide crystallographic society celebrated the 100th anniversary of the discovery of the X-rays by W. C. Röntgen.

Organizers and sponsors of the conference were the Russian State Committee of High Education, Commission on X-ray Analysis of Raw Materials of the Mineralogical Society, Scientific Council of the Russian Academy of Natural Sciences, St. Petersburg University, Belgorod State Technological Academy for Building Materials and Russian Agency for the Support of Fundamental Research. Prof. S. K. Filatov (St. Petersburg) served as Chairman of the Organizing Committee.

The scientific program of the conference addressed the following topics:

- crystal chemistry of layer silicates;
- determination and crystallochemical analysis of crystal structures;
- application of X-ray powder diffraction to problems of technology and synthesis of crystalline materials under various p-T-c conditions;
- methodology, data bases and instrumentation for X-ray studies.

The volume of abstracts of conference papers presented 103 items including five plenary lectures, 21 oral contributions, 65 posters and 12 reports. Among the most

remarkable contributions, the following ones deserve to be explicitly mentioned:

Recalling Prof. V. A. Frank - Kamenetsky (S. K. Filatov), Crystallochemistry of Transformations in Layer Silicates (E. A. Goilo), The Contribution of Electron Diffraction to the Structural and Lattice Crystallography of Minerals (B. B. Zvyagin), Crystallochemistry of Minerals with OT₄-oxocentric Polyions (S. K. Filatov et al.), Quartz Materials and the Problem of the Manufacture of Quartz Oscillators (H. Bradaczek), On Occupation of Crystallographic Positions as an Information Source on Structural Faults in Minerals (O. V. Frank - Kamenetskaya), On Directional Recrystallization Mechanisms in Chrysotile (G. A. Krinari et al.), New Varieties of Layer Silicates with Tubular Morphology (A. Gorshkov et al.), Thermal Strains and Transformations of Layer Silicates by High-Temperature X-ray Diffraction Analysis (E. N. Kotelnikova), Changes of Mica Molecular Structure Induced by Heating and Mechanical Deformation (V. A. Liopo et al.), The History and Present Status of the Computerized X-ray Diffraction Phase Analysis in the Soviet Union (E. K. Vasiliev and V. G. Evsyunin), The Determination Technique and Genetic Interpretation of Chlorite in Poor Formed Rocks (G. A. Krinari), X-ray and Electron Diffraction in Ecology Monitoring (V. A. Liopo and V. V. Voyna), The Crystal Structure of a New Mineral Odintsovite (R. K. Rastsvetarva et al.), On Crystal Structure of Orthorhombic Lazurite (A. N. Sapozhnicov et al.) and On Two Types of Modulation of Hauyne Crystal Structure (A. N. Sapozhnicov et al.)

Some 70 participants from Russia, Ukraine, Belorussia, Moldova, Germany and other countries took part at the conference.

E.K. Vasiliev, Irkutsk

XRF and XRD Workshop

Havana, Cuba, 7-8 November 1995

Siemens Analytical X-ray Systems, one of the world leaders in manufacturing X-ray instruments, organized a two-day workshop on X-ray fluorescence (XRF) and X-ray diffraction (XRD) techniques in Havana, Cuba in November 1995. About 45 participants per day, including scientists and managers from universities and research institutions with R & D profile, had the opportunity to see the latest products in instrumentation, hardware and software from Siemens, and the applications of X-ray powder diffraction in industrial areas such as cement, metallurgy, and pharmaceutical.

The workshop opened new ways for cooperation between the scientists involved in XRF and XRD at different institutions and industry, in order to interchange experience, to carry-on research projects and to facilitate joint efforts to up-date and up-grade their analytical equipment. This workshop was held in November 7

- 8, 1995, exactly 100 years after the discovery of X-rays by Wilhelm Conrad Röntgen.

Hector Novoa de Armas, C. Q. F., La Habana, Cuba

Conference "The Application of X-rays in Science and Technology"

Irkutsk, Russia, 16-23 November 1995

The conference has been dedicated to the 100th anniversary of the discovery of X-rays and to the 150th anniversary of the birthday of its discoverer, the first Nobel - Prize winner for physics, German physicist Wilhelm Conrad Röntgen. The conference was organized by the Faculty of Physics of the University of Irkutsk with Prof. Yu. V. Agrafov serving as chairman of the organizing committee.

Among 26 lectures and reports delivered at the conference which reflected the contemporary level of the fundamental and applied X-ray diffraction and spectroscopy research as performed in Siberia, the following were of top importance: The History of the Discovery and Development of X-ray Investigations (E. K. Vasiliev), Direct Methods for the Study of Electron Density Deformations (L. I. Kleshchinsky), The Sun's X-rays (A. A. Golovko), The Crystal Structure of Hauyne (V. G. Evsyunin et al.), A Study of the Melt-Crystal System, being in Thermodynamic Equilibrium, with the Help of X-ray Dispersion (Yu. V. Agrafov and T. E. Ushakov), A Study of Magnetostriction of Ferromagnetics by X-ray Powder Diffraction Technique (V. A. Domyshev et al.), The Approximation of X-ray Diffraction Line Profiles by Splines (G. L. Ivanov), The Application of XRF Analysis in Geology (A. G. Revenko), The XRF of Elements with Small Atomic Numbers (G. V. Pavlinsky) and An Application of X-ray Spectral Analysis for Monitoring of Environmental Pollutions (A. N. Smagunova and T. N. Gunicheva).

E. K. Vasiliev, Irkutsk

The International Schools and Conference on X-ray Analytical Methods AXAA 96.

Sydney, Australia, 18-25 January 1996

The international Schools and Conference on X-ray Analytical Methods (AXAA 96), organized by the Australian X-ray Analytical Association, was held at the University of New South Wales in Sydney from the 18th to 25th January 1996. Altogether it attracted nearly 200 delegates of whom approximately 60 were from outside Australia. For someone who is used to either academic or single discipline conferences, AXAA gatherings are unique. In the first place they bring together both X-ray diffractionists and X-ray spectroscopists. They also attract delegates from a variety of disciplines and variety of backgrounds including universities, research establishments, government and in-

dustry. This year the perspective was broader than usual both because of the marking of 100 years of analytical X-rays, and the strong showing of overseas presenters. The result was a very interesting and stimulating forum where one was able to experience a wide range of experimental and theoretical problems ranging from

practicalities of carrying out X-ray analysis in a remote mining community, to the design of the latest high resolution powder diffractometer at the Photon Factory in Japan.

The first three days of AXAA 96 were devoted to the Schools and Workshops program, half of which were XRD based and other XRF based. The Schools program, which took up the first day and half, was designed specifically for young people who are relatively new to these fields and as such the lectures were pitched at a level that assumed limited knowledge and experience of the field. Three connected themes were introduced over the three sessions of the XRD School: X-ray Physics, Crystals and Diffraction, and X-ray Diffractometry. Altogether there were 11 lectures in these sessions attended by both beginning and experienced X-ray analysts, and without exception the lectures were of a very high standard and contained lots of practical tips that would be difficult to find in any reference text. Deane Smith's animated lecture on Escher and symmetry in crystallography lingers in my memory as does Hideo Toraya's overview of XRD in materials analysis and the latest developments in diffraction instrumentation.

Following the Schools program, eleven three hour workshops were held on more specialized areas of X-ray analysis. Six of those were on powder diffraction. Rietveld Analysis figured prominently in this program first with a theory based workshop presented by Ian Madsen, Daniel Louer, Tony Cheetham and Mark Raven. This was followed up by a "hands-on" workshop convened by Ian Madsen and Kate Hawkins in which each of the 35 participants had the luxury of an individual PC to experience the thrills and frustrations of the Rietveld analysis. Special thanks go out to Jaane Steer of the UNSW Applied Geology department for organizing the PCs. Other XRD workshops included,

- An ICDD Workshop run by Ron Jenkins and Deane Smith,
- Modern Techniques and Diffraction under Non-Standard Sample Environment, by Dudley Creagh, Leif Gerward and Cliff Ball,
- X-ray Line Broadening by Bob Cheary, Daniel Louer and Walter Kalceff,
- Grazing Incidence X-ray Analysis by Ray Goehner and Mike Eatough.

Once delegates had consumed the Schools and Workshop program, they entered the second phase of AXAA 96, the half day Centenary Symposium celebrating 100 years of X-ray analysis. The afternoon started with Keith Norrish recounting the early days of XRF analy-

sis in Australia, and progressed through to Tony Cheetham's splendid lecture on the history of X-rays and how far we have come since Roentgen's discovery. The day was capped off with a Welcome Function on a pleasantly warm Summer's evening (in January!) to mark the start of phase three of AXAA96, the Conference program.

The problem with reporting any conference is that it is impossible to discuss anything in-depth without appearing biased or of limited vision. What I recount here are my personal memories of the Conference which I recall without recourse to notes. I therefore apologize in advance for the many speakers and presenters I have overlooked. I derived great pleasure listening to Daniel Louer discussing how far we had come with conventional X-ray sources. Hiroo Hashizume's plenary lecture was also memorable because it made me aware of a new method of analyzing thin film interfaces using the diffusely reflected X-rays in the vicinity of the critical angle. Paul Fewster made me appreciate that with modern well designed high resolution instruments there are still many things to be discovered by investigating diffraction from near perfect crystals. On the last day of the conference Karl Mauser revealed that

X-ray mirrors for converting a diverging X-ray beam from a conventional X-ray tube to a parallel X-ray beam are readily available (at a cost?). The advantages of parallel

beams, taken for granted by synchrotron users, can now be exploited on the laboratory based diffractometers. Special mention must also be made of the poster by Michael Mantler and colleagues from the Technical University of Vienna for their excellent poster on the Thermal Expansion of Titanium Boride, measured using a new high temperature camera, which won the poster prize.

All in all I was glad to be at AXAA 96. It was an excellent conference for networking with established colleagues and developing new colleagues. The manufacturer's exhibits were harmoniously blended into the delegates meeting area, and the congenial atmosphere and general ambience of the University of New South Wales campus made for a very friendly and relaxing meeting. I will certainly be attending the next AXAA meeting wherever and whenever it is held.

Bob Cheary, Sydney

WHAT'S ON

4-6 June 1996

Fourth European Conference on Residual Stresses, Cluny, France. Contact: Societe Francasise de Metalurgie et de Materiaux - SF2M-ECRS4, 1 rue Paul Cezanne, 75008 Paris, France. FAX +33-1-49-53-71-00.

3-9 August 1996

Satellite Meeting on Powder Diffraction associated with the IUCr Congress on Crystallography and Denver X-ray Conference, Denver, Colorado, USA. Contact: Prof. Paul K. Predecki, Dept. of Engineering, University of Denver, CO 80208, USA. FAX +1-303-871-4450, DENXCON@DIANA.CAIR.DU.EDU

8-17 August 1996

17th IUCr General Assembly and International Congress of Crystallography, Seattle, Washington,

USA. Contact: Prof. R. F. Bryan, Dept. of Chemistry, Univ. of Virginia, Charlottesville, VA 22903, USA.

25-28 May 1997

5th European Powder Diffraction Conference (EPDIC-5), Parma, Italy. Contact: Prof. G. Artioli, Dipartimento di Scienze della Terra, Universita di Milano, Via Botticelli 23, I-20133 Milano, Italy. Tel. 39-2-236 98320, FAX 39-2-706 38681, ARTIOLI@IUMMIX.TERRA.UNIMI.IT

25-28 August 1997

17th Conference on Applied Crystallography, Cieszyn, Poland. Contact: Prof. H. Morawiec, Institute of Physics and Chemistry of Metals, University of Silesia, Bankowa 12, 40-007 Katowice, Poland. Tel. +(48 32) 59-69-29, FAX +(4832) 59-96-05, DANA@USCTOULI.CTO.US.EDU.PL

NEWS FROM ICDD

The International Centre for Diffraction Data is pleased to announce the awarding of four Crystallography Scholarships for 1996. Mr. Patrick M. Len, University of California, Davis, USA; Mr. Aaron Oakley, St. Vincent's Institute of Medical Research, Australia; Ms. Liat Shimoni, Fox Chase Cancer Center, USA; and, Mr. Hongwu Xu, Princeton University, USA, have been designated recipients by the ICDD Scholarship Award Selection Committee. Patrick Len's research centers on the theoretical development of a holographic technique to determine atomic crystal structure from electron or fluorescent X-ray diffraction data. Aaron

Oakley is exploring structural and functional aspects of glutathione S-transferases by X-ray crystallography and computer graphics techniques. Liat Shimoni is determining the crystal structures of two enzymes: porphobilinogen synthase and ubiquitin carboxyl-

terminal hydrolase. Hongwu Xu is studying "High Temperature TEM and X-ray Diffraction Studies of the Order-Disorder Transition in Beta-Eucryptite".

At the Fall meeting of ICDD, Deane K. Smith was elected by the Board as a Distinguished Fellow. Deane has contributed greatly over the past 30 years as a scientist, Board Member (Chairman), Grantee, Journal Editor and

principal teacher of our very successful XRD Clinics. His international contributions to conferences such as in Australia, Denver, and Egypt are well recognized by his colleagues. His current activity with the program data bank has saved many scientists from "re-inventing the same software". Deane recently retired from Penn State after 27 years as a Professor of Mineralogy.

On December 20, 1995 Chairman G. G. Johnson, Jr. announced to the staff and ICDD members that Dan Richardson had submitted his resignation as the General Manager of ICDD as of January 1, 1996. Dan brought new perspective to the Centre and leaves the organization on a strong financial footing. The Executive Committee has begun the process of locating a new General Manager.

On February 22, 1996 the results of the recent election were announced: Chairman: Robert L. Snyder; Vice Chairman: R. A. Young; Technical Committee Chairman: T. C. Huang; Members-at-large, Board of Directors: Camden R. Hubbard, James A. Kaduk; Thomas N. Blanton, Cyrus E. Crowder, and Charlotte Lowe-Ma will continue as Members-at-large. G. G. Johnson, Jr. fills the position of Past Chairman.

On March 21, 1996 the ICDD Board of Directors elected Julian Messick as Treasurer and Ron Jenkins as Secretary and General Manager.

The ICDD X-ray Clinics will be held in June 1996 at the International Centre for Diffraction Data Headquarters in Newtown Square, Pennsylvania. The ICDD Clinic on X-ray Powder Diffraction will be held in two week-long sessions as follows: Fundamentals of X-ray Powder Diffraction (June 3-7, 1996), Advanced Methods in X-ray Powder Diffraction (June 10-14, 1996). The ICDD Clinic on X-ray Fluorescence Spectrometry will be held June 17-21, 1996.

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Ludo Frevel, ICDD Representative

MAILING LIST FOR NEWSLETTERS

If you would like to be added to the mailing list for Newsletters of the Commission on Powder Diffraction of the IUCr or you have changed your address, please contact the CPD Secretary, Dr. Daniel Louer at the above mentioned address.

CALL FOR CONTRIBUTIONS TO THE NEXT CPD NEWSLETTER

This issue of the CPD Newsletter has been produced by the Czech and Slovak Crystallographic Association, edited by Jaroslav Fiala with considerable technical assistance of Radomir Kuzel from Prague. The next issue will be edited by Dr. LYNNE McCUSKER to appear in October 1996. She would greatly appreciate contributions from readers on matters of interest to the powder diffraction community, e.g. meeting reports, future meetings, developments in instruments, techniques and computer programs and news of general interest. Please, send articles and suggestions directly to her (address shown below).

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