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Meeting Report

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Report on the Satellite Meeting on Powder Diffraction SMP93, Hangzhou, China, 31 August–3 September 1993

The meeting was held in one of the most beautiful locations in China. The West Lake of Hangzhou is famous for carp, mist and poetic recitations. If the meeting itself could not offer these qualities, it did offer a wide range of powder diffraction topics and an almost inexhaustible supply of green tea. The local committee, chaired by Professor Rong-Guo Ling, made a superb job of every aspect of organization, including the presentation of the programme and abstracts, the lake cruise and the final exceptional culinary feast at the University of Hangzhou.

The meeting was organized under the auspices of the IUCr Commission on Powder Diffraction, a major function of which is to raise awareness of powder diffraction around the world and to highlight some of the most significant recent work in the field. The conference was split into four major categories: structural studies; instrumentation standards and data bases; non-ambient and time-dependent powder diffraction; and the characterization of materials.

After an opening address by the Governor of the Province, the President of the University and the Chairman of the Commission on Powder Diffraction, the meeting got under way with a fascinating talk about the history of powder diffraction in China where, despite many difficulties, much high-quality work is being carried out, including analytical work on the La–Ba–CuO system by Dong *et al.* [*Phys. Rev. B* (1988), **37**, 5182–5185]. Also highlighted was the multilayer work using synchrotron radiation of Wong *et al.* [*J. Appl. Phys.* (1973), **73**] and, more recently, the work of Qiu-zi Cong [*Acta Cryst.* (1993), **A49**, 23–26]. Powder diffraction in China is still mostly a research tool and is not widely used for analysis in industry. There are some 1200 powder instruments in China, many of which have been constructed locally in Dandong and Beijing. Interest in the technique is growing and it was encouraging to see so many groups within China producing work of such high quality.

The first session was entitled Structural Studies Based on Powder Data. C. Gilmore (Glasgow, Scotland) described in brief the maximum-entropy/maximum-likelihood estimates method and recent successes with structure determination using the program *MICE*. The technique is able to utilize identified structural fragments in those cases where there might be insufficient phasing information for conventional Fourier calculations and is relatively stable when using data of different resolutions. The method builds up phasing trees, the nodes of which represent phase choices; these choices are then selected by likelihood procedures. The method is particularly suitable for powders since it uses all reflections, including those that are closely overlapped. Future directions should include efficient codes and improved handling of overlapped data.

C. Giacovazzo (Bari, Italy) described the application of direct methods to solving crystal structures from powder diffraction data. In outlining the program *SIRPOW92*, he emphasized the need to include weak reflections and the weighting of data to carry overlap status into phasing equations. The way in which the program treats single and overlapping reflections is also new; and intensity modifications of strongly overlapped regions is possible after initially phasing on the more reliable low-angle well resolved data. The method has been shown to be successful on a wide variety of organic and inorganic materials.

C. Howard (ANSTO, NSW, Australia) outlined the application of maximum-entropy methods (MEM) to the calculation of electronic and nuclear densities (X-ray and neutron) for CeO_2 and rutile [*J. Appl. Cryst.* (1993), **26**, 159–165]. The early results are encouraging and the method has advantages over Fourier methods because the electron density is positive everywhere. Future directions could see the MEM applied in whole-pattern refinement.

M. Tremayne (St Andrews, Scotland) further promoted the MEM/likelihood approach with examples and showed that Monte Carlo methods can also be successful, particularly with molecules containing rigid chemical groups. She described the structure solution of two previously unknown structures, $LiCF_3SO_3$ and p-$CH_3C_6H_4SO_2NHNH_2$. The treatment of peak overlap is thought to be superior using this method, hence its application to *ab initio* structure determination should be even greater for more complex structures.

X. Cheng (Hangzhou, China) described a metastable Fe_3O_4 phase, refined by the Rietveld method and R. Hill (CSIRO, Victoria, Australia), reporting the CPD Round Robin II results for the Rietveld refinement of monoclinic SrO_2, noted the optimum conditions required for both data collection and refinement strategy (*e.g.* peak/background ratios of at least 50:1). As expected, the refined positions of the oxygen atoms had higher standard deviations and a much greater spread for the X-ray data than for the neutron data. The thermal parameters were not reliably determined for the majority of the X-ray data refinements, especially in those cases where the observations-to-parameters ratio was less than about 5:1.

The next session concentrated on applications of the above methods with an emphasis on the complementarity of laboratory X-rays, synchrotron radiation and neutron diffraction data. A. Fitch (ESRF, Grenoble, France) elegantly illustrated how both synchrotron radiation and neutron diffraction can be essential to a correct solution in some applications: the case study was $KUO_2PO_4.3H_2O$, which was expected to be tetragonal, but with the high-resolution synchrotron data proved orthorhombic (0.2% difference in a and b cell edges). Moreover, the key 011 reflection, which defines the correct space group, has very weak X-ray intensity. In addition, the ordering of benzene saturated in ZSM-5 zeolite was impossible without the combined advantages of the two radiations. These examples showed the genuine complementary nature of X-rays and neutrons.

M. Estermann (ISIS, RAL, England) complemented an excellent talk at the main Congress on incorporating prior chemical knowledge in structure solution problems with some practical examples. He described some typical problems and possible solutions that arise from the applications of traditional single-crystal methods to powder data: Wilson plots giving negative B values; the equipartition of closely overlapping reflections can give a fallacious artificially acentric statistical distribution. He also described the use of the fast interactive Patterson squaring method coupled with DLS calculations in *SAPO*-40. The structure of cloverite was given as an example of the powder method failing – it was finally solved using a single crystal with synchrotron radiation.

P. E. Werner (Arrhenius, Stockholm, Sweden) demonstrated the usefulness

of high-resolution electron microscopy (HREM) to distinguish closely related phases (high-pressure W_3O_8 with a common *c*-axis length). He also noted that in a high-pressure WO_2 sample 97% of the X-ray scattering came from W – this results in space-group-definition problems, with the correct space group increasing the number of data from 163 to 280 reflections but with only two significant observations in the additional data.

C. J. Sparks (Oak Ridge National Laboratory, USA) talked about sample granularity which led to a marked decrease in the low-angle intensities, hence increasing the *B* factors. The use of anomalous (resonance) edge wavelengths was described, which gives information about site preferences for similar atomic number (*Z*) elements in alloys. These effects differ markedly for high- and low-*Z* elements and for the latter these effects will be absorbed in the refined thermal parameters.

Two well presented talks by students then followed: D. Gascoigne (Birkbeck, London, England) and N. Sudo (Tokyo Institute of Technology, Japan) on the *ab initio* structure determinations of $Zr(OH)_2SO_4.3H_2O$ and the high-pressure form of Mg_3BN_3, respectively. T. Larsson (Uppsala, Sweden) completed the first day's programme with the analysis of ZrOAl–O phases, which absorb a considerable amount of hydrogen. By using neutron diffraction from three proposed disordered models and absorption measurements, he concluded that the near saturation phase could be described as $\Sigma r_5Al_3O_{0.5}D_{4.5}$.

The second day began with a review by R. Jenkins (ICDD, USA) of databases, new ICDD systems and search strategies, indicating the future potential to store full patterns *via* CD-ROM media.

M. McMahon (Edinburgh, Scotland) then described how the limitations in size and resolution in high-pressure diffraction studies have been successfully addressed by the use of (synchrotron-radiation) angle-dispersive techniques and image-plate area detectors. This method has applications in the study of groups III–V, II–VI and IV semiconductors. Anomalous dispersion was used to distinguish similar atoms in these materials. The data have very good signal/noise and can pinpoint very subtle effects, such as a new cinnabar phase in CdTe. The data collections were carried out at SRS Daresbury. In the future it should be possible to have pressure cells handling pressures >50 GPa, more accurate intensities by better preferred-orientation and microstrain broadening models and even more intense beam sources such as the ESRF or dedicated national very high flux synchrotron sources.

T. Blanton (Kodak, NY, USA) reviewed the status of Standard Reference Materials (including the instrument sensitivity standard SRM1976) and then presented the case for silver behenate as a low-angle calibration standard. With several strong reflections from Cu $K\alpha$ radiation, accurate cells were determined from a round-robin measurement. Only questions of thermal expansion, surface chemical stability and the ability to make a permanent sample need be resolved.

Several talks then addressed instrumentation and methods: Qui-zi Cong (Lanzhou, China) on the formalism of sample-tilting diffraction for examining layered samples. D. Bertelmann (Siemens, Germany) spoke on the advantages of the Siemens Hi-Star 2D area-detector/laser-beam alignment system. This is able to analyse 200–300 μm regions of sample using short (minutes) exposures, with examples given of rubbers, polymers and wool (but not silk!), H. Toraya (Nagoya, Japan) talked about symmetric and asymmetric profile diffraction theory with conventional focusing and pseudo-parallel beam geometries (using CeO_2 examples) and H. J. Bunge (Clausthal, Germany) discussed the advantages of a Siemens D500 linear position-sensitive detector (over a scintillation counter) for texture analysis. These included faster more accurate background measurements and easier separation of overlapping peaks. The examples given included polymers, CuZnAl shape memory alloys and glass ceramics.

In a session devoted to non-ambient and time-dependent diffraction, Y. Fuji (Tokyo, Japan) described the three SR stations set up for high-pressure (diamond-anvil cell) studies at the Photon Factory, covering low, room and high temperatures. High pressure in the halogen materials induces an atomic from a molecular solid at pressures from 5 to 85 GPa, which can be studied by using synchrotron radiation with the imaging plate. This was the first work carried out in the area and has led to the development of a very successful technique.

T. Yamanaka (Osaka, Japan) reviewed his laboratory's time-resolved computer-aided measurement and control system (with PSD) and its application to the dehydration studies of $Mg(OH)_2$ and $Ca(OH)_2$. He also described the study of the GeO_2 quartz-to-rutile-type transition and the lattice deformation of Si and Ge semiconductors using pulsed laser irradiation with 50–100 ns data-acquisition times.

J. Loveday (Edinburgh, Scotland) described an improved opposed-anvil Paris–Edinburgh pressure cell for neutron radiation (up to 10 GPa), with examples including non-metallic hydrides (*e.g.* ND_3), ice VIII and squaric acid. Future pressure cells for neutron diffraction should approach 25 GPa. I. Grey (CSIRO, Melbourne, Austrailia) outlined Rietveld constrained refinement as applied to defining the phase diagram of the non-stoichiometric metal oxides M_3O_5, pseudobrookite and M_2O_3; the complementarity of the X-ray and neutron studies was highlighted. I. Shannon (St Andrews, Scotland) showed an analysis of urea/alkane inclusion compounds.

Continuing and expanding upon his plenary lecture at the main congress, W. David (RAL, England) outlined his high-pressure low-temperature studies of buckyball C60 using the High-Resolution Neutron Powder Diffractometer at ISIS. Two models are found at low temperature based on data collected between 5 and 272 K, with the more stable having the larger volume; thermodynamic calculations and models predict that at 260 K and 2.6 kbar the two structures should interconvert. This has been one of the areas where powder neutron diffraction has made a very significant contribution.

D. Hausermann (ESRF, Grenoble, France) described the advances in high-pressure diffraction using the enhanced brightness at the ESRF with the image plate detector. An X-ray image intensifier allows 2K x 2K pixel readout in 5 s and 512 x 512 pixels in 2 μs. A cell has been developed for 2 x 2 x 2 mm samples held at 60 kbar with pressure gradient <0.3 kbar across the sample.

The third day's symposium concerned characterization of materials. H. J. Bunge (Clausthal, Germany) reviewed the effects of texture in powder diffraction, including the *ODFO* analysis program. Y.-M. Wang utilized the studies of nitrided iron and steel to illustrate the identification of microscopic/macroscopic stress in relation to the depth profile, noting that dislocation theory is not yet complete. I. Langford (Birmingham, England) used studies of ZnO to illustrate the need for complete information in using Williamson–Hall plots. The cylindrical particle model (rather than spherical) gave linear relationships of height and diameter against temperature of preparation from the oxalate dihydrate. The analysis concluded that stacking faults exist in the material.

J. Schneider (Munich, Germany) concluded in a comparison of X-ray and neutron refinements that, as the latter has less aberrations, it should give a higher-accuracy result.

C.-I. Kuo (Shanghai, China) addressed the stability of simultaneous equations in Zevin's standardless phase-analysis method. M. Jarvinen (Lappeenranta, Finland) outlined texture-factor models involving Gauss and March functions, concluding that the March function is not

appropriate for cubic-system powders. The orientation error present in radial distribution functions was addressed by the final speaker, J.-Z. Chen (Tianjin, China).

The general ambience of the meeting was excellent and left a lasting and very favourable impression upon the delegates. A big vote of thanks was also given to J. I. Langford for his excellent work as Chairman of the Programme Committee.

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Crystallographers

*This section is intendeed to be a series of short paragraphs dealing with the activities of crystallographers, such as their changes of position, promotions, assumption of significant new duties, honours etc. Items for inclusion, subject to the approval of the Editorial Board, should be sent to The Executive Sectretary, 2 Abbey Square, Chester CH*1 *2HU, England.*

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Dr **Isabella Karle** has been awarded the 1993 Bower Award and Prize for Achievement in Science by the Franklin Institute.

New Commercial Products

Announcements of new commercial products are published by the Journal of Applied Crystallography *free of charge. The descriptions, up to 300 words or the equivalent if a figure is included, should give the price and the manufacturer's full address. Full or partial inclusion is subject to the Editor's approval and to the space available. All correspondence should be sent to the Editor, Dr A. M. Glazer, Editor* Journal of Applied Crystallography, *Clarendon Laboratory, University of Oxford, Parks Road, Oxford OX*1 *3PU, England.*
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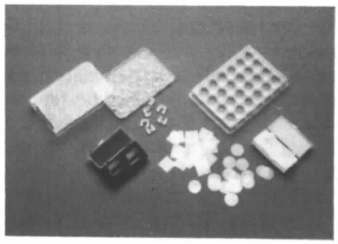
Supplies for Sitting- and Hanging-Drop Crystallization

Hampton Research is offering **presiliconized square and circle cover slides** for hanging-drop crystallization. The siliconized cover slides provide an optimal hydrophobic surface for hanging-drop vapor-diffusion crystallization. Siliconized cover slides are available in 22 mm squares and circles for the popular Linbro plate as well as 18 mm circles for the smaller footprint Linbro, Falcon and Costar plates for crystal growth.



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Books Received

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The following books have been received by the Editor. Brief and generally uncritical notices are given of works of marginal crystallographic interest; occasionally, a book of fundamental interest is included under this heading because of difficulty in finding a suitable reviewer without great delay.

Growth of crystals. Vol. 19. Edited by *E. I. Givargizov* and *S. A. Grinberg*. Pp. viii + 202. New York: Plenum Publishing Corporation, 1993. Price $95.00. ISBN 0-306-18119-3. This is a translation of the original Russian text published by the Institute of Crystallography of the Russian Academy of Sciences in 1989. It is based on invited papers from the Seventh All-Union Conference on the Growth of Crystals and the Symposium on Molecular-Beam Epitaxy, held in Moscow in November 1988. It contains four papers on growth of crystals from the vapor, three on growth of crystals from the melt, four on growth of crystals and films from fluxes (including single crystals of high-T_c superconductors in the La–Sr–Cu–O, Y–Ba–Cu–O and Bi–Sr–Ca–Cu–O systems, described by Dem'yanets, Bykov and Mel'nikov) and four on the structure of crystals and films relative to growth conditions.