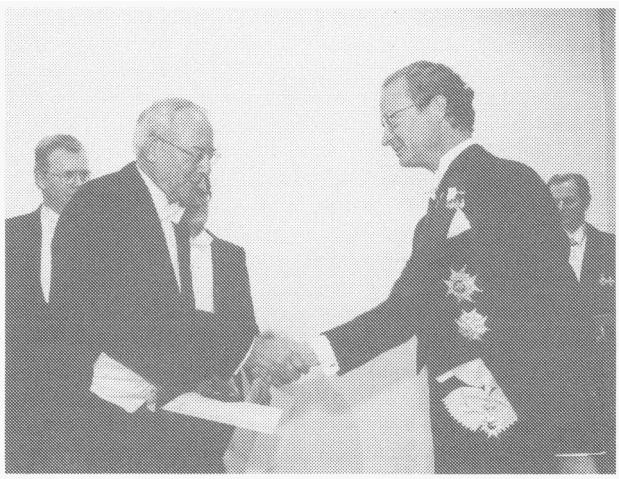
COMMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

NEWSLETTER No. 14, MAY 1995

The Aminoff Prize to HUGO RIETVELD



Dr Hugo M Rietveld receives the Aminoff prize from the hands of His Majesty the King Carl XVI Gustaf.

The Royal Swedish Academy of Sciences has awarded the Aminoff Prize of 1995 to Dr Hugo M Rietveld for his outstanding contribution to the analysis of powder diffraction data. Hugo Rietveld received the prize on the Anniversary Meeting of the Academy in Stockholm on March 31, 1995. The prize includes a gold medal designed by Gregori Aminoff and a sum of money. The prize ceremony was followed by a dinner at the Karlberg Castle in the presence of Their Majesties the King and the Queen.

The Aminoff Prize

The Aminoff Prize is named after Gregori Aminoff who was born on February 8, 1883 in Stockholm and died in 1947. He was elected member of the Royal Swedish Academy of Sciences in 1933. His family is descended from a Russian officer who joined the Swedish army in 1612.

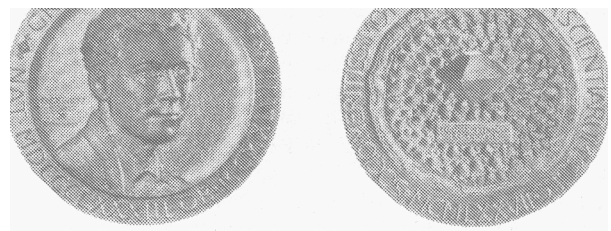
Gregory Aminoff had two careers - as an artist and as a scientist. While still a schoolboy he was interested in minerals and collected them. The famous Arctic explorer Adolf Erik Nordenskiöld, who was Professor of Mineralogy at the Museum of Natural History, gave him free access to the mineral collection there. In 1905, at the age of 22, Aminoff had already received bachelor's degree and published two minor papers.

It was then that he switched to painting, enrolling in a famous art school and joining "The Young", a group of young painters who were to become very well known. Aminoff was successful and painted in Paris, London and Italy. He studied for a period with Henri Matisse.

In 1914 when the First World War broke out his career as an artist ended. He resumed his scientific studies and gained his doctorate in 1918. In the same year he introduced X-ray crystallography into Sweden. He became Professor of Mineralogy at the Museum of Natural Sciences in 1923.

On 21 September 1950 Aminoff's widow Birgit Broomé-Aminoff wrote her will, providing for the establishment of a fund, the Professor Gregori Aminoff Memorial Fund, to be administered by the Royal Swedish Academy of Sciences. An annual prize, the Gregori Aminoff Prize, was to be awarded for theses published in the field of crystallography. It was to be possible for several winners to share the prize.

The prize is intended to reward a documented, individual contribution in the field of crystallography, including areas concerned with the dynamics of the formation and dissolution of crystal structures. Some preference should be shown for work evincing elegance in the approach to the problem.



The Aminoff medal

Prizewinners:

1979	Prof P P Ewald	USA
1980	No award this year	
1981	Sir Charles Frank	UK
1982	Prof Gunnar Hägg	Sweden
1983	Prof J M Robertson	UK
1984	Dr David Harker	USA
1985	Prof André Guinier	France
1986	Prof E F Bertaut	France
1987	Prof Otto Kratky	Austria
1988	Dr Isabella L Karle	USA
1989	Prof Arne Magnéli	Sweden
1990	Prof Jack Dunitz	Switzerland
1991	Sir David Philips	UK
1992	Prof M M Woolfson	UK
1993	Prof Clifford G Shull	USA
1994	Prof Michael G Rossman	USA
1995	Dr Hugo M Rietveld	The Netherlands

The Aminoff Symposium

An Aminoff Symposium was organized by the Academy on March 30, 1995, in honour of Dr Rietveld. All Nordic countries were represented among the 65 participants who listened to invited speakers from Sweden and abroad lecturing on the past, the present, and the future of the Rietveld Refinement Method.

Hugo Rietveld himself gave a retrospective view, and Alan Hewat, ILL presented the early history from Harwell and Grenoble. Josh Thomas, Uppsala University, talked about the first application to X-rays and Bill David, RAL, described time-of-flight Rietveld refinement.

Swedish contributions covered structure solution, synchrotron radiation, magnetic structures, metal hydrides and high T_c superconductors. The final talk on future development of the Rietveld method was given by Juan Rodrigues-Carvajal, Laboratoire Léon Brillouin, Saclay.

R.T.

Chairmans report

It has been another active year for the CPD during 1994. We have been involved in the organisation of two very successful powder diffraction meetings in Russia and South Africa, and have assisted with the preparation for three meetings scheduled for 1995 in England and Slovakia. Planning also continued for the CPD's involvement in the IUCr Congress in Seattle in 1996, together with the associated Satellite Meeting on Powder Diffraction in Denver. The results of Part II of the Rietveld Refinement Round Robin were published, and a grant was provided in support of the continued development of the World Directory of Powder Diffraction Programs. Direct communication with powder diffractionists continued with the issuing of two CPD Newsletters in April and October.

The International Workshop on "Advanced Powder Diffraction Techniques in Mineral and Materials Processing" was held in Pretoria, South Africa, in October, 1994, and was co-organized by the Crystallographic Society and Mineralogical Association of South Africa. CPD member Dr Lynne McCusker was an invited speaker and served as Chairman of the Scientific Program Committee, member Dr Ian Langford was also an invited speaker, while CPD Chairman Dr Rod Hill contributed to the Program Committee. The meeting was an outstanding success, with 85 participants gaining valuable experience from a series of carefully crafted lectures, tutorials and hands-on workshop sessions on all aspects of powder diffraction analysis, including sessions presented by the ICDD.

The International Conference on "Powder Diffraction and Crystal Chemistry" was held in St Petersburg, Russia, in July 1994, co-organised by the Russian Association Powder Crystallography, the Dept of Crystallography of St Petersburg University, and the CPD. Lectures and posters were presented to 165 participants from 20 countries, including a workshop run by the ICDD, and a 211-page book of abstracts was produced. St Petersburg's strong past and current links with crystallography, dating from Fedorov's discovery of the 230 space groups in 1890, along with its geographic beauty, provided a very appropriate and delightful backdrop for a most successful conference.

The CPD is lending its support to the EPDIC-IV meeting in Chester in July 1995 and to the International Conference on X-ray Powder Diffraction Analysis of Size/Strain, Macrostress and Texture in Slovakia in August 1995 - CPD Member Dr Jaroslav Fiala and the CPD Chairman are serving on the Programme Committees for both of these meetings, Dr Fiala as Chairman for the Slovakia meeting. Preparations for a CPD-organised Micro-symposium on powder diffraction at the 1996 IUCr Congress and General Assembly in Seattle have commenced, including nominations for a session Chairman and possible topics. Similarly, arrangements were put in place for a Satellite Meeting on Powder Diffraction to be held in Denver in August 1996, in conjunction with the 45th Meeting of the Denver X-ray Conference.

The CPD remained active in the execution of its existing projects and in the initiation of new ones. Part I1 of the Rietveld Refinement Round Robin Project was published in the Journal of Applied Crystallography, 27, pp 802-844. This part of the project provided outcomes of the analysis of Rietveld refinements of the monoclinic zirconia crystal structure based on X-ray and neutron powder diffraction data collected on a 'standard' sample with a wide spectrum of instruments located in 12 countries around the world. Work on Part JII has now commenced - this part will focus on a multivariate analysis of the results obtained from the zirconia refinements.

The CPD confirmed its ongoing support for the World Directory of Powder Diffraction Programs, co-produced by CPD member Prof Deane Smith and Dr Syb Gorter from the Netherlands, through the provision of a grant for the purchase of computer hardware. Organisation of a new round robin project on Phase Quantification by Diffraction Methods, coordinated by Deane Smith, has continued with a call for expressions of interest and suggestions for the type of tests and samples that might be used. Also, arrangements have continued for a survey of methods and models in use for the determination of crystallite size and microstrain from powder diffraction data under the auspices of a Task Group co-chaired by Dr Fiala and Prof Bob Snyder of the USA.

I encourage you to participate in any or all of these CPD projects, to provide us with advice and topics for meetings, to recommend projects in which the CPD might become involved, and to offer contributions for the next Newsletter (due around October of this year).

Rod Hill, CPD Chairman

IUCr COMMISSION ON POWDER DIFFRACTION

Chairman: Rod J. Hill, Division of Minerals, CSIRO, Box 312 Rosebank MDC, Clayton, Victoria 3169, Australia. Tel: +61-3-545-8602; FAX: +61-3-562-8919; E-mail: RODH@MINERALS.CSIRO.AU.

Secretary: Daniel Louër, Laboratoire de Cristallochimie, Chimie du Solide et Inorganique Moléculaire, Universite de Rennes 1,35042 Rennes, Cedex, France; FAX: +33-99-38-34-87; E-mail:DANIEL.LOUER@UNIV-RENNES1.FR

Members

R. (Bob) J. Cernik, Daresbury Laboratory, Daresbury, Warrington, WA4 4AD, UK; FAX: +44-925-603174 or 100; E-mail: CERNIK@DARESBURY.AC.UK.

Dave E. Cox, Physics Department, Brookhaven National Laboratory, Upton, NY 11973, USA; FAX: +1-516-282-2739; E-mail: COX@BNLX7A.NSLS.BNL.GOV.

Jaroslav Fiala, Department of Metallurgy, Central Research Institute SKODA, Tylova, 46, 31600 Plzen, Czech Republic; FAX: +42-19-773-3889

Lynne B. McCusker, Institut fur Kristallographie und Petrolographie, ETH Zentrum, CH-8092 Zurich, Switzerland. FAX: +41-1-632-1133; E-mail: LYNNE.MCCUSKER@KRISTALL.ERDW:ETHZ.CH).

Shao-Fan Lin, Test and Computation Centre, Central Laboratory, Nankai University, Tianjin 300071, PR China; FAX: +86-22-350-1555); E-mail: FENGCB@BEPC2.IHEP.AC.CN.

Deane K. Smith, 239 Deike Building, Department of Geosciences, The Pennsylvania State University, University Park, PA 16803, USA; FAX: +1-814-863-7845; E-mail: SMITH@VAXl.MRL.PSU.EDU.

I.G. Roland Tellgren, Institute of Chemistry, Uppsala University, Box 531, S-75121 Uppsala, Sweden; FAX: +46-18-320355; E-mail: RTE@KEMI.UU.SE.

Hideo Toraya, Ceramics Research Laboratory, Nagoya Institute of Technology, Asahigaoka. Tajimi 507, Japan; FAX: +81-572-27-6812; E-mail: TORAYA@CRL.NITECH.AC.JP.

ICDD Representative

Ludo K. Frevel, 1205 W. Park Drive, Midland, MI 48640, USA; FAX: +1-610-325-9823.

Consultants

J. Ian Langford, School of Physics and Space Research, Birmingham University, P. **O.** Box 363, Birmingham, B15 2TT, UK; FAX: +44-121-414-4709; E-mail: LANGFOJI@NOVELLl.BHAM.AC.UK.

Ray A. Young, School of Physics, Georgia Institute of Technology, Atlanta, GA 30332-0430, USA; FAX: +1-404-853-9958; E-mail: PH268RY@GITVM1.GATECH.EDU.

Gunnar Hagg and the Guinier-Hagg-type X-ray powder diffraction cameras

Stig Rundqvist
Department of Inorganic Chemistry, University of Uppsala, Sweden

Introduction

Gunnar Hagg (1903-1986), professor of Inorganic Chemistry at the University of Uppsala 1936-1969, is probably best known internationally as one of the pioneers in chemical crystallography and one of the founders of structural solid state chemistry. He was very active within the IUCr, serving as vice-president 1951-1957 and also as a member of the Union's Commissions on Crystallographic Apparatus and on "Structure Reports". Less well-known are perhaps his achievements as inventor and constructor of various pieces of laboratory equipment. The present article describes Hagg's contributions to the development of Guinier-type X-ray powder diffraction cameras, which have retained their ro1e as indispensible tools in Swedish solid state chemistry research for more than 40 years.

The first Guinier-Hagg camera

When Hagg started his work as professor in Uppsala, the laboratories contained no equipment for X-ray diffraction work, and he had to start from scratch to create the necessary facilities. In spite of all difficulties, and the even more severe conditions following the outbreak of the Second World War, Hagg managed to build up an excellent X-ray diffraction laboratory, which was fully operational in the early 1940:s. For powder diffraction work, focussing cameras of the Seemann-Bohlin type [1,2] were used.

At this time, Hagg was familiar with the methods for monochromatizing and concentrating divergent X-rays by means of properly ground and elastically bent single crystals [3,4], and he was also aware of the combination of a Johansson-type monochromator with a focussing powder diffraction camera made by A. Guinier [5,6].

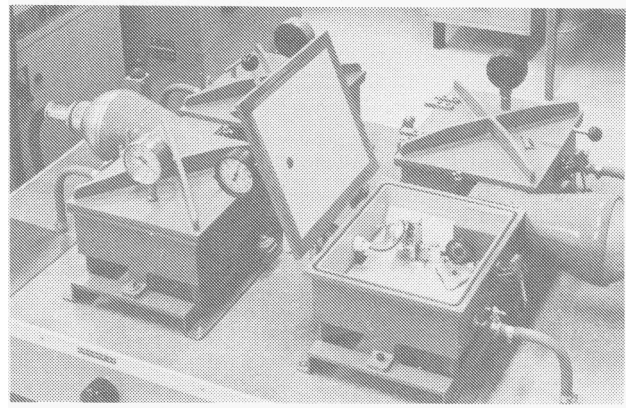
In 1949, Hagg visited Guinier in Paris and became very favourably impressed by the performance of Guinier's camera. Soon afterwards, the first powder diffraction camera based on the Guinier principle was built in Uppsala. The whole camera setup was fixed to a solid bottom plate placed in a vacuum chamber. X-rays from a normal-focus Cu-anode tube entered the vacuum chamber through a beryllium window. The X-ray tube was mounted in the "point focus" position: the focal line on the anode lying perpendicular to the cylinder axis of the monochromator crystal. This was an a-quartz crystal, cut in the form of a thin rectangular plate with the surface inclined some 3*from the 10-loplane, ground cylindrically to a curvature of 500 mm radius and clamped between two cylindrical brass blocks to a final radius of 250 mm. When the monochromator was aligned for maximum intensity, the reflected beam contained $CuK\alpha_1 + CuK\alpha_2$ radiation.

The apparatus was arranged for transmission of the X-rays through the sample. The sample holder consisted of a circular brass plate with a central hole admitting the X-rays from the monochromator. The hole was covered by adhesive tape onto which the sample powder was sprinkled. The sample holder was rotated by a simple electric motor device.

The cylindrical film cassette of radius 40 mm and covering diffraction lines from $\theta = 0°$ to 45° was placed in the so-called subtraction mode. This caused the positions of the α_1 and the α_2 lines on the films to become interchanged on going from low to high diffraction angles, the two lines coinciding at around θ = 15'. Film measurements and film shrinkage corrections were made using methods and devices developed by Hagg already for the Seemann-Bohlin recordings [7]. Automatic corrections for shrinkage were made by printing a scale divided in 0.1 mm on the films before development. Measurements of the films were carried out with the aid of the printed scale using a projection comparator with suitable magnification.

The "second generation" Guinier-Hagg cameras

The construction and performance of the first Guinier-Hagg camera (denoted G 1) was a great success, and the building of additional cameras followed in rapid succession as the result of an increasing demand for powder diffraction facilities. The "second generation" cameras were assembled in two rectangular vacuum chambers placed on opposite sides of a copper radiation X-ray tube. Each chamber was provided with a monochromator, sample holder and cassette setup fixed onto a bottom plate, similar to the G 1 arrangement. Each of the bottom plates could be removed and replaced by another bottom plate. One of these was provided by a "two-storied" rectangular sample holder (which could be rocked back and forth by a simple device) and a "two-storied" cassette, making it possible to record diffractograms of two samples simultaneously. A camera arrangement placed on a second bottom plate was provided with an aluminium monochromator with double curvature, as described in [8].



A set of four "second generation" Guinier-Hägg cameras, Dept. of Inorg. Chem., Uppsala 1965

The current research programs put an increasingly heavy load on the available powder cameras, and efforts were therefore made to reduce the exposure times for the recordings. The "subtraction" geometry used was actually one step in that direction: the intensities in the "crucial" θ region around 15° being enhanced by the α_1/α_2 overlap. Another step in the same direction was the attempt to introduce double curvature monochromators. The aluminium monochromators [8] actually produced a 16-fold intensity gain as compared with the quartz monochromators, but unfortunately, plastic deformation in the crystals impaired their X-ray optical properties so that sharp lines and α_1/α_2 resolution could never be achieved.

From about 1955 and onwards, Hagg was very much occupied by writing a textbook in inorganic chemistry, and he did not resume his activities as a constructor of scientific apparatus until a few years before his retirement. In the meantime, much of the equipment in the X-ray laboratory was replaced by modern commercial instruments, but the Guinier cameras were retained almost unchanged except for some minor improvements. For instance, the introduction of fine-focus X-ray tubes and turning them from point- to line-focus

orientation made it possible to record diffractograms with pure $K\alpha_1$ radiation and to reduce the exposure time considerably.

The 'third generation" Guinier-Hägg cameras

When Hagg resumed his work on Guinier-type cameras, he planned firstly to redesign his cameras for work at room temperature, and secondly, to construct cameras for work at high temperatures and high pressures.

As regards the cameras for ordinary purposes, Hagg designed the new model so as to fit Philips commercial X-ray generators. The camera was to be placed on a bracket support close to the X-ray tube window, with the X-ray path in a vertical plane. A prototype was manufactured in the departmental workshop, and a machine for grinding quartz or germanium lamellae for monochromator crystals was also built at the same time. The camera prototype was finished in 1967. Commercial production was taken up by a Swedish company, and the camera was included under the label XDC-700 in Philips analytical equipment program [9]. The monochromator crystals were ground by an optician in Uppsala using the machine mentioned above, and the final adjustments and checks of the cameras were made at the Inorganic Department before delivery.

The high-temperature camera

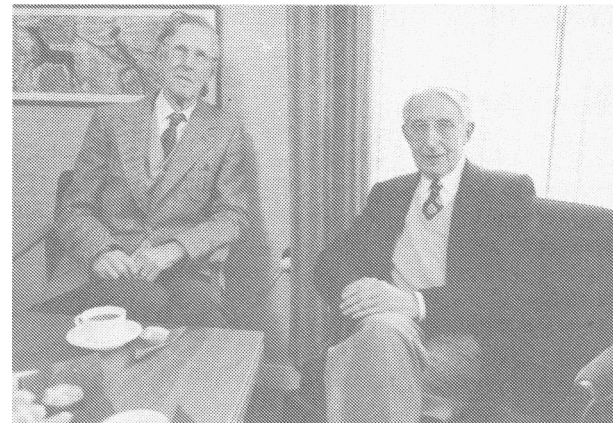
For the high-temperature camera, a germanium monochromator (reflecting plane (111)) was chosen rather than a quartz monochromator in order to gain intensity. The monochromatic beam entered a chamber (which could be evacuated or filled with an inert gas) through a beryllium window at the bottom, passed the flat horizontal sample, and left the chamber, together with the diffracted rays, through a long slit, covered with a beryllium sheet. The film cassette was placed above this slit on top of the chamber, and the films could be changed without breaking the vacuum or interrupting the heating run. The sample, placed on a horizontal fibre mat support, was surrounded by a tantalum element heated resistively, and a set of radiation shields, cut in a suitable manner so as to admit the passage of the X-rays. The temperature of the sample was read pyrometrically through a quartz glass window.

The camera construction which was described in detail in [10] could operate successfully up to temperatures near 2000ºC, with a quality of the film recordings approaching that for the ordinary room-temperature cameras. In later years, the camera was provided with an automatic temperature control via a thermocouple arrangement.

The high-pressure camera

For high-pressure diffraction work, Hagg adopted ideas described by *i.a.* McWhan and Bond [11] to construct a modified Guinier-type camera. The load from a hydraulic ram was applied on the sample contained in a pressure gasket of amorphous boron, placed between Bridgman anvils of sintered tungsten carbide. In contrast to earlier constructions, where the press frame and the anvil assembly could be oscillated, these were stationary in Haggs apparatus, while the X-ray system (including X-ray tube, monochromator and film holder) could be oscillated about the sample. $MoK\alpha$ radiation from a Johann-type [12] germanium monochromator was used. The sample powder, generally mixed with sodium chloride for pressure calibration purposes, was placed in a 0.25 mm axial hole in an amorphous boron disk of 3 mm diameter and 1 mm height. Pressures, monitored by an ordinary load cell, could be applied up to some 15 GPa.

An initial version of the camera was finished in 1976 [13]. Problems of maintaining the specimen in a fixed position during pressure application prompted a redesign of the film cassette. The new film holder was placed cylindrically around the sample, transforming the camera into a hybrid between the Guinier and the Debye-Scherrer types [14]. Diffraction lines could now be recorded on both sides of the primary beam, making it possible to apply corrections for sample misalignment.



Gunnar Hagg and André Guinier at the Dept. of Inorg. Chem. in Uppsala 1983.

Concluding remarks

In May 1985, The Royal Swedish Academy of Sciences and 1'Académie des Sciences de 1'Institute de France arranged a French-Swedish conference in Stockholm, entitled "Advances in Powder Diffraction Crystallography" in honour of Professor André Guinier and Professor Gunnar Hagg. The two prominent scientists, having developed a good friendship over the years, both provided oral contributions to the conference. In the conference proceedings, Hagg's paper, entitled "Powder Diffraction of X-Rays: Personal Reminiscences 1927-1957" [15], included descriptions of some of his earlier activities related in the present paper.

In retrospect, it seems indeed very satisfying and fortunate that Hagg could attend the conference and, as honorary participant together with his colleague and friend Guinier, receive this well-earned recognition of outstanding achievements in powder diffraction techniques, before his death the following year.

References

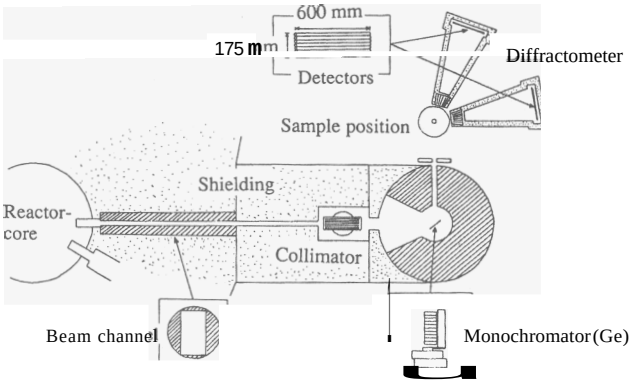
1. Seemann, H., Ann. Phys. (4) 59,455 (1919).
2. Bohlin, H., Ann. Phys (4) 61,421 (1920).
3. DuMond, J.W.M. and Kirkpatrick, K.A., Rev. Sci. Instr. 1, 88 (1930).
4. Johansson, T., Naturwiss. 20,758 (1932).
5. Guinier, A., Compt. rend. (France) 204, 1115 (1937).
6. Guinier, A., Ann. Phys. 12, 161 (1939).
7. Hagg, G., Rev. Sci. Instr. 18, 371 (1947).
8. Hagg, G. and Karlsson, N., Acta Crystallogr. 5,728 (1952).
9. Hagg, G. and Ersson, N.O., Philips Bulletin of Analytical Equipment, Sept. 1971.
10. Hagg, G., Ersson, N.O., Rudenholm, G. and Sellberg, B., J. Appl. Cryst. 12, 221 (1979).
11. McWhan, D.B. and Bond, W.L., Rev. Sci. Instr. **35,** 626 (1964).
12. Johann, H.H., **Z.** Phys. 69, 185 (1931).
13. Hagg, G., Engstrom, I. and Törmä, B., in "High-pressure Science and Technology", Vol. 1, p. 890; Timmerhaus K.D. and Barber, M.S., Eds., Plenum, New York (1979).
14. Ersson, N.O., Chem. Scr. 26A, 65 (1986).
15. Hagg, G.,Chem. Scr. 26A, 3 (1986).

A NEW POWDER NEUTRON DIFFRACTOMETER AT THE JEEP II REACTOR AT KJELLER IN NORWAY

Bjørn C. Hauback, Helmer Fjellvåg and Olav Steinsvoll
Institutt for Energiteknikk, P.O. Box 40, N-2007 Kjeller Norge

A new high-resolution powder neutron diffractometer, PUS, is under installation at the 2 MW JEEP II reactor at Institutt for Energiteknikk at Kjeller in Norway. Special emphasis is put on designing a flexible instrument where the utilization of useful neutrons are maximized. At a low-flux reactor, like JEEP II with a central flux of about $2.5 \cdot 10^{13}$ neutrons cm^{-2} s^{-1}, great efforts are required to optimize the relationship between intensity (signal/noise ratio) and resolution. In that respect, the most important components of the diffractometer system are collimators, the monochromator, the detector and the shielding.

A schematic drawing of PUS is shown in the Figure. A new beam channel with height 90 mm and width 50 mm has been installed in the reactorshielding from the reactor tank to the first collimator (α_1). The beam channel can be filled with water, to work as a beam shutter, or during experiments, by a noble gas, to reduce scattering by air and thus increase the neutron flux on the monochromator. A three-section Soller collimator system (manufactured by Riser National Laboratory in Denmark) will be used as the α_1-collimator. The sections with widths 50 mm and 15', 30' or completely open collimation, will provide what we here call high resolution, medium resolution and medium intensity, and high intensity modes, respectively. An open collimator corresponds to about 60' natural collimation. The collimator system will be mounted on motorized translational and rotational tables.



A schematic drawing **of** the new neutron powder diffractometer at Kjeller.

The flux at the sample position will be enhanced by using a vertically focussing germanium monochromator. The incoming white beam of height 90 mm will be focused on a sample of a typical height 30-40 mm. Until very recently, bulk Ge crystals were usually plastically deformed to introduce the optimum mosaic widths (with respect to intensity and resolution). However, for bulk Ge crystals the mosaic distributions tend to vary significantly from crystal to crystal, and are in most cases not acceptable for high resolution applications. Instead, Axe et a1 [I] at Brookhaven National Laboratory bent thin wafers of Ge (each 0.4 mm thick) which were bonded together to give composite germanium plates of thickness 8 mm. Compared to the traditional bulk Ge monochromator, the individual pieces of the composite Ge monochromator show reproducible behaviour, both with respect to uniformity and the anisotropic mosaic distribution, with a small vertical and a larger horizontal mosaic distribution. A composite monochromator of this type is under production in collaboration with Riser National Laboratory. The practical work is presently carried out at Risø, based on the description and experience from Brookhaven.

The Ge wafers are cut with the (511) planes parallel to the reflecting surface and according to a vertical [0,1,-1]axis in order to enable reflections from different (hkk) planes, e.g. (311) and (711), by a simple rotation of the monochromator-unit. In addition, the monochromator take-off angle can be varied by a rotation of the monochromator shielding, (see drawing). With take-off angles in the range 55-loo", and reflections from Ge (hl1), h=3,5,7; monochromatic neutrons with wavelengths in the range 0.75-2.6 A.,will be available for experiments.

The mechanical construction of the diffractometer (manufactured by GMI in Grenoble) consists of two detector-units and a sample module. The complete unit moves on air cushion pads, and allows a free rotation of the detector-units around the sample table. The floor is of Riser-type with 1 mm formica plates on a 20 mm epoxy sublayer. The sample module consists of three rotating tables, one for each of the detector-units, with absolute encoders, and one for rotation of the sample itself, to reduce preferred orientation effects. A rotating oscillating collimator system will be mounted in front of each of the two detector-units to suppress scattering from the sample environment. The shielding of the detector-units consists of self-supporting constructions of so-called neutrolene plates from GMI (8 weight% B_2O_3). The thickness of neutrolene will be at least 200 mm everywhere. The inner walls, made of aluminium, will be covered with Gd_2O_3 containing paint.

Each of the two detector-units will contain seven commercially available position sensitive ^{3}He detectors (from Reuter&Stokes) stacked on top of each other. The single detectors have diameter 25 mm and effective length 600 mm. The neutron event positions are determined by the digital charge division method for the individual detectors. The data from each of the seven detectors will be handled separately, and finally summed by the software. With a sample - detector distance of 1.6 m, each detector-unit will cover 20" in 2θ. The resolution of the detectors is guaranteed to be better than 5 mm. A complete powder pattern of 120" will be collected by moving both detector-units together in three steps.

Different sample environments will be available. Temperatures ranging from 8 K to approximately 1600 K will be obtained by means of a Displex cooling system and a furnace.

All parts of the instrument will be controlled by software running on a PC-486 computer using the OS-9000 operative system. A HP-712 workstation will be connected to the instrument with graphics and programs for data treatment, Rietveld refinements and general crystallographic calculations.

According to the present plan the diffractometer should be put in operation during summer 1995, commissioned during the autumn and available for users at the end of the year. As described above, priority has been given for optimizing those parts of the instrument which are important for the intensity.

If you have any further questions, please feel free to ask us.

REFERENCES

[1] J.D. Axe, S. Cheung, D.E. Cox, L. Passell, T. Vogt and S.Bar-Ziv.
J. Neutron Research (1994) 85-94.

MEETINGS

Report on the 3rd Regional Czecho-Slovak Conference on Powder Diffraction. Liptovsky Mikulas, 3-6 October 1994

25 crystallographers from six countries gathered in a modem campus of the Military Academy Liptovsky Mikulás near a great lake (Liptovská Mara) in the midst of the romantic Slovak mountains Liptov to discuss a number of interesting problems regarding X-ray diffraction analysis of the structure of polycrystalline materials: solid state amorphisation induced in the Fe-Ti system by mechanical deformation (S.Enzo), texture development in electrotechnical nonoriented silicon steels (M. Cernik), microdeformations in zinc oxide thin films (P. Sutta, Q. Jackuliak, V. Tvarozek and I. Novotny) etc. A volume of proceedings (multilangual, 86+IIpp, ISBN 80-8040-013-X) was provided to attendees of the conference upon registration. The social program included a conference banquet, an excursion into the mountains and a visit to the department of physics (incl. X-ray Diffraction Laboratory) of the host Academy. These annual conferences are intended to serve as a communication pool for powder diffraction crystallographers in the region of the Czech and Slovak Republics with international participation. Thanks to the endeavour of the local organizers (Dr. Sutta, Head of the Department of Physics of the Academy), beautiful surroundings and a deep concern of the participating crystallographers, the conference was a big success. **Jaroslav Fiala**

SOUTHERN AFRICA POWDER DIFFRACTION WORKSHOP - PDSA-94 An International Workshop on Advanced Powder Diffraction Techniques in Mineral and Materials Processing, Pretoria, South Africa, 24 - 27 October 1994

The seeds for PDSA-94 were sown at the Bordeaux IUCr Congress, when Prof. Jan Boeyens, Chairman of the SA National Committee for Crystallography, presented to the CPD a convincing case for holding a powder-diffraction workshop in South Africa. The Commission agreed to support Prof. Boeyen's proposal in principle, but it was not until the Beijing Congress that a decision was taken to go ahead with the meeting. In the event, the workshop was organised by the SA Crystallographic Society and the Mineralogical Association of SA, in collaboration with the CPD. The venue chosen by the Local Committee, Chaired by Prof. Gert Kruger, was the Geological Survey of South Africa, on the outskirts of Pretoria - partly known as 'Jacaranda City'. All the workshop literature, from the first circular to the proceedings, displayed a colourful print of Pretoria's jacaranda-lined streets, but no photograph could do justice to the sea of electric blue which greeted participants when they arrived in the city.

The main aim of PDSA-94 was to introduce crystallographers and mineralogists in southern Africa to modern powder diffractometry in a practical way, through lectures, tutorials, demonstrations and discussion sessions. The Programme Committee, chaired by Dr Lynne McCusker, managed to persuade several leading authorities in different fields of powder diffraction to assist with the workshop - if any persuasion were needed! This was made possible by generous contributions from a long list of sponsors, including the ICDD, various firms and organisations within SA, and equipment manufacturers with agencies there, and of course the IUCr, who provided the usual bursaries for young scientists.

Participants, guest speakers and organisers met on the eve of the workshop for a buffet reception in the mineralogical section of the Transvaal Museum, which provided an opportunity for leisurely viewing of many exceptional specimens from a country noted for the variety of its mineral species. The first day of the workshop, Monday 24 October, was devoted to invited lectures on basic topics, by way of introduction to the tutorial sessions. After an introductory overview, Dr Ron Jenkins opened the proceedings with characteristic enthusiasm, giving talks on instrumentation and databases for powder diffraction. Dr Lynne McCusker followed, with a lucid talk on the basic principles of indexing. She contrived to make this complex topic appear deceptively simple and, by way of a check that her talk had been understood, insisted that members of the audience indexed data for a gallophosphate for themselves! Then Dr Christian Barlocher dealt with calculating a powder diffraction pattern and the first day concluded with the first session on applications - phase identification and quantitative analysis by means of 'traditional' methods - by Dr Julius Schneider. During the evening Dr Faan le Roux invited the guest speakers to visit the AEC of SA's Safari-1 reactor and powder-diffraction laboratories, followed by a (licensed!) buffet in the garden of Preller House, a former Boer farmstead. For the remaining three days of the workshop, each half-day session started with a lecture and this was followed by parallel small-group tutorials. Sessions concluded with various demonstrations of software, equipment and techniques. Tuesday was devoted to crystal structures, with talks by Dr Barlocher on the Rietveld method and Dr McCusker on structure determination from powder data. Within minutes of the end of the last afternoon session, all were transported to the CSIR Recreational Site for a traditional Braai - a sheep-roast, to the uninitiated.

Despite the long, hard day on Tuesday, all arrived promptly at 9 o'clock on Wednesday for Prof. Bill David's overview of the use of fixed-wavelength and time-of-flight neutrons in powder diffraction, illustrated by some of the exciting new materials studied in recent years. This talk was not only memorable for its content, but also for the use of the latest PC technology in its presentation. The afternoon lecture, by Dr Schneider, was on quantitative analysis by means of the Rietveld method. The final day was devoted to imperfect structures and samples. Dr Ian Langford gave a talk on how to extract information on sample microstructure from powder diffraction data and, in the afternoon, Prof. Hans Bunge covered the influence of texture in powder diffraction. Preferred orientation is a nuisence in most applications of powder diffraction, but Prof. Bunge demonstrated the importance in materials science of mapping the texture of polycrystalline samples. There were no fewer than 30 one-hour tutorial sessions, mostly conducted by the guest speakers on topics related to the content of the invited lectures, but including instruction from Dr Dieter Biihmann on current practice in characterising clay minerals by means of powder data. The somewhat unusual feature of having five parallel tutorial sessions, slotted into a programme of lectures and demonstrations, resulted in a very intensive four days, but the format was highly successful, in that a high attendance of the 80 or so participants was maintained to the end. The success of the workshop was due in no small measure to the first-rate facilities at the Geological Survey and to the efforts of Dr Buhmann in ensuring its smooth running from start to finish.

Regarding the success of PDSA-94, there can be no

doubt. The main objective of providing crystallographers in southern Africa with hands-on experience of modern techniques in powder diffraction was certainly achieved. Hopefully, this will be the first of many such workshops in SA and the neighbouring countries.

J. Ian Langford

Report on the Symposium "Powder Diffraction", Osaka, 25 November 1994

The Annual Meeting of the Crystallographic Society of Japan was held at Osaka University in November 24-26, 1994. A joint symposium between Japan and Australia on powder diffraction using synchrotron and neutron radiations was held as one of the microsymposiums. It was timely because Australia has succeeded in constructing the multi-purpose powder diffractometer called BIGDIFF at BL-20B experimental station in Photon Factory (PF), Tsukuba.

Six invited lectures were presented as follows:

"The multi-purpose diffractometer on the Aust. Natl. Beamline at the PF" by Prof. D. Creagh of Univ. New South Wales.

"Initial experiments with BIGDIFF on beamline 20B at the PF and prospects for use in powder diffraction application studies" by Dr. D. Cookson of Aust. Natl. Beamline Facility, PF.

"Analysis of powder diffraction collected on the Aust. Natl. Beamline at the PF' by Dr. B.J. Kennedy of Univ. Sydney.

"Current powder diffraction research using synchrotron radiation in Japan" by Dr. H. Toraya of Nagoya Inst. Tech.

"Neutron powder diffraction in Aust. materials research" by Dr. C.J. Howard of Aust. Nucl. Sci. Tech. Org., and

"Applications of TOF neutron powder diffraction to high-T, super-conductors'' by Dr. F. Izumi of Natl. Inst. Res. Inorg. Mater.

The symposium was organized by Prof. H. Hashizume of Tokyo Inst. Tech. It involved a half day programme, and more than 60 people attended. **Hideo Toraya**

WHAT'S ON

5-30 June 1995

ICDD X-ray Clinics

ICDD Clinics on X-ray Powder Diffraction

June 5-9 Fundamentals of X-ray Powder Diffraction

June 12-16 Advanced Methods in X-ray Powder Diffraction

ICDD Clinics on X-ray Fluorescence Spectrometry

June 19-23 Fundamentals of X-ray Fluorescence

June 26-30 Advanced Methods in X-ray Fluorescence

Contact: Theresa Maguire , Internat. Centre for Diffraction Data, 12 Campus Boulevard, Newtown Square, PA 19073-3273, Phone: (610) 325-9823, E-mail: Maguire@ICDD.COM

12-16 June 1995

Rontgen Centenary Congress: 1895-1995, 100 Years of X-rays" to be held at the International Convention Centre, Birmingham, UK. Contact: Carol Curran, The Royal College of Radiographers, 14, Upper Wimpole Street, London W1M 8BN; Tel. (44) 171 935 5726, Ext. 2131, Fax (44) 171 487 3483.

10-15 July 1995

EPDIC IV. Chester College, UK. The 3-day meeting will cover all aspects of powder diffraction, with particular emphasis on structure solution and refinement, dynamic studies of structures under change and the industrial importance of the technique. The scope of the meeting will be as broad as possible and will be covered by a series of plenary and invited speakers. There will be comprehensive poster sessions in all areas of powder diffraction. Contact: Dr R. J. Cernik, Daresbury Laboratory, Daresbury, Warrington, WA4 4AD, UK. Fax: +44 925 603195; E-mail: cernik@daresbury.ac.uk

16-20 July 1995

Ab-Initio Structure Determination Using Powder Diffraction Techniques. Oxford, UK. Speakers will include Christian Baerlocher, Dave Cox, Andy Fitch, Chris Gilmore, Lynne McCusker, Kenneth Shankland, Gerard Bricogne, Bill David, Clive Freeman, Rod Hill, John Newsome, Devinder Sivia, Tony Cheetham, Michael Estermann, Carmello Giacovazzo, Daniel Louër, Rene Pescher and Per-Erik Werner. Topics to be covered include optimal data collection strategies; autoindexing; Bayesian methods; intensity extraction; direct methods; Rietveld refinement; maximum entropy/likelihood; prediction; computer modelling. Places limited to about 100. Contact: Dr W I F David. (e-mail: wifd@isise.rl.ac.uk) or Kenneth Shankland.

20-22 July 1995

RSS 95-RS. Rietveld Summer School in Russia A Rietveld Summer School is being organized by Russian scientists, interested in crystallography and powder diffraction, in cooperation with the IUCr Commission on Powder Diffraction. This 3-day short course will deal with the principles and techniques of Rietveld analysis to avoid or overcome problems in the use of it. The well-grounded beginner and the moderately experienced user, including students and young scientists, should find the course valuable. The program will include lectures, tutorials and 'hands-on' experience on PC computers. More detailed information, registration and grant application forms can be obtained from: Dr V.V. Chemyshev, Chemistry Dept. Moscow State University, 119899 Moscow, Russia. (Fax 7-(095)-9390898.
E-mail: vladimir@struct.chem.msu.su)

23-28 July 1995

American Crystallographic Association Meeting. Montreal, Quebec, Canada. Contact: Dr. **Y.** LePage, Program Chairman, NRC of Canada, Chemistry Department, Ottawa, Ontario, KIA OR6 Canada. Tel: +I-613-993-2527; Fax +I-613-952-1275; E-mail: yvon@iecems.lan.nrc.ca.

6-11 August 1995

16th European Crystallographic Meeting. Lund, Sweden. Contact: Dr. Ake Oskarsson (Chairman), Department of Inorganic Chemistry 1, Chemical Center, Lund University, P.O. Box 124, 5-221 00 Lund, Sweden. Tel: +46-46-108102; E-mail: ake.oskarsson@inorgkl.lu.se.

21-25 August 1995

International Conference on X-ray Powder Diffraction Analysis of Size/Strain, Macro-stress and Texture. Liptovsky Mikulás, Slovakia. Contact: Dr. J. Fiala, Department of Metallurgy, Central Research Institute SKODA, Tylova 46, 31600 Plzen, Czech Republic. Fax: +42-19-773-3889.

4-8 September 1995

Sixth National Conference on X-ray Diffraction and the Hundredth Anniversary Meeting for Röntgen's Discovering X-ray, Tianjin, China, supported by Physics Society of China, Crystallography Society of China and Chemistry Society of China. The conference is organized by Nankai University and X-ray Analyses Society of Tianjin. The 5-day conference

will cover all aspects of X-ray diffraction, X-ray fluorescence and crystallography. Some academicians of Academia Sinica will be invited to give main lectures. There will be seven microsymposia beside plenary. Contact: Prof. Lin Shao-Fan or Prof. Yao Xinkan, Central Laboratory, Nankai University, 300071, Tianjin, PR Chinca. Tel: +86-22-350-1380; FAX: +86-22-350- 1555;E-mail: FENGCB@BEPC2.IHEP.AC .CN

12-21 September 1995
4th Oxford Summer School on Neutron Scattering. Oxford, UK. Sponsored by IUCr. Contact: Professor B.T. M. Willis, Chemical Crystallography Laboratory, 9 Parks Road, Oxford, OX1 3PD, UK. Fax: +44 865 272960.

5-7 October 1995
Annual Meeting of the Crystallographic Society of Japan, Nagoya Univ., Japan. Contact: Dr. T. Yamane, Dept. Biotech., Faculty of Engin., Nagoya Univ., Huro-cho, Chikusa, Nagoya 464-01, Japan FAX (81)-52-789-3223, E-mail: a40439a@nucc.cc.nagoya-u.ac.jp.

22-24 November I995
Second Conference of the Asian Crystallographic Association (AsCA'95), Bangkok, Thailand. The conference is intended as a forum of all the fields of crystallography in Asia. A variety of crystallographic contributions are welcomed. Contact: Prof. Y. Ohashi, Dept. Chem., Tokyo Inst. Tech., Ohokayama, Tokyo 152, Japan, FAX (81)-3-3720-6206, E-mail: yohashi@chem.titech.ac.jp.

3-9 August 1996
Satellite Meeting on Powder Diffraction associated with the IUCr Congress on Crystallography and Denver X-ray Conference, Denver, Colorado. Contact: Prof. Paul K. Predecki, Department of Engineering, University of Denver, Denver, CO 80208, USA. Fax: +1-303-871-4450; E-mail: 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, Department of Chemistry, University of Virginia, Charlottesville, VA22903, USA

NEWS FROM ICDD

NEW GENERAL MANAGER OF ICDD

Julian Messick, Corporate Secretary and General Manager of ICDD has announced his retirement after 29 years of service with the International Centre. Julian has served the ICDD in many capacities and has held the Secretary/General Manager position for the past 13 years.

The Centre takes great pleasure in announcing that effective 1 January 1995 Daniel C. Richardson has assumed this position. Dan is a graduate of the U.S. Naval Academy, retiring from the Navy with the rank of Rear Admiral after thirty years of service. Dan holds a B.S. in Naval Science, an M.A. in Mechanical Engineering and a M.S. in Business Administration in addition to numerous post graduate studies. Dan completed his academic training at Harvard University. He is the former Chief of Staff of the Valley Forge Military Academy and College.

Under the new administration, the Centre looks forward to continued growth and cooperation with its Scientific and Business Associates. Julian Messick will continue as a corporate liaison for the immediate future.

GRANTS-IN-AID PROGRAM

Each year the ICDD extends financial support in the form of grants-in-aid to a limited number of institutions worldwide. These grants are for the provision of high quality, demonstrably useful X-ray powder diffraction patterns, In 1994, the ICDD supported 29 proposals in the total amount of $203,600. Recipients of these awards represented 13 countries. For further information regarding the program, please contact:

Theresa Maguire
Manager, Grants & Education Adiministration
International Centre for Diffraction Data
12 Campus Boulevard
Newtown Square, PA 19073-3273
Telephone: 610-325-9814 FAX: 610-325-9823
E-mail: Maguire@ICDD.COM

CRYSTALLOGRAPHY SCHOLARSHIPS AWARDS

The ICDD Scholarship Award Committee received 43 applications by the deadline date of October 31, 1994. During the month of November, each of the five members of this committee independently reviewed all 43 applications. The awards are granted to:

L. R. MacGillivray:	Univ. of Missouri-Columbia, USA
Faculty Advisor:	Jerry L. Arwood
Research Topic:	Noncovalent Interactions and Crystal Engineering: Understanding the Solid-state Self-Assembly of Molecules and Ions Isolated from Liqid Clathrate Media
Kevin F. Peters:	Northwestern University, USA
Faculty Advisor:	Jerome B. Cohen
Research Topic:	Small Particle Melting Phenomena
Arturas Vailionis:	Royal Inst of Technology, Sweden
Faculty Advisor:	Anders Flodstrom
Research Topic:	Structural Characterization of Bi2 Sr2 Ca (n-1) Cu (n) O (y) thin films with n22

COOPERATING ORGANIZATIONS

ICDD is pleased to announce that the Ceramic Society of Japan has been elected as a Cooperating Organization of ICDD. There are now 14 such organizations. Cooperating Organizations designate one of their members as a representative to ICDD. This representative is afforded full voting authority by the Centre on issues that may require a ballot. Also, each representative is encouraged to represent the respective society's views on topics that may come forth for discussion.

TRAINING FACILITIES AT HEADQUARTERS

A non-working x-ray room has been furnished with a donated diffractometer, various cameras and sample preparation facilities. Students at the ICDD clinics will be able to perform many of the actual "hands-on" that they requested in their evaluation of the Clinics over the past five years.

Ludo Frevel, ICDD Representative

CALL FOR CONTRIBUTIONS TO THE NEXT CPD NEWSLETTER

The next issue of the CPD Newsletter will be edited by Prof HIDEO TORAYA to appear in October of 1995. He 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 directlv to him (address on V 2). **R. Telleren. Editor. CPD Newsletter 14**