01-Instrumentation and Experimental Techniques (X-rays, Neutrons, Electrons)

The newly developed equipment for micro-region analysis was designed to be installed with a micro-pinhole and with an imaging plate(IP;Fuji Co.Ltd.) readout system. Even though the equipment is placed in a vacuum chamber to avoid air scattering, a diffraction pattern at different crystal orientations can be obtained without opening the chamber. Micropinholes with diameters of 5 and 10 μm were prepared and set just after the collimator. The distance between the pinhole and the sample is 7 mm, and the detecter using IP covers from -30 to 165 degrees in two-theta range with camera radius of 100mm. This appratus with a 10μm pinhole was initially applied to olivine(Mg_2SiO_4) included in a thin section of meteorite, and also to micrometer-sized aluminum grains on a semiconductor material.

PS-01.03.08 COMPUTER-AIDED CRYSTAL ORIENTATION

By Y.J. Jiang*, X. N. Wang, L. Z. Zeng, Department of Applied Physics, Beijing Polytechnic University, Beijing, 100022, China.

There are many methods for adjiustment of the single crystals. Among them, the Laue method is the most general one for the determination of orientation and symmetry of crystals (Wood, Crystal Orientation Manuel,Columbia University, New York, 1963). In order to reduce the time required for conventional Laue back-reflection method, we have developed COMPUTER-AIDED CRYSTAL ORIENTATION(CACO).

Procedure and main points of CACO are briefly described in the following.

1. Mount the crystal to be oriented on a goniometer, take a Laue photograph.
2. According to the spatial arrangment and intensity of spots on Laue photograph, select a major spot (X', Y') which is not only a strong reflection but one through which many zones pass,predefine the corresponding Miller indices to be (h'k'l'). Move (X', Y') to the centre of Laue photograph, transform other spots. Display the transformed Laue photograph on screen.
3. Calculate positions and intensities of Laue spots,simulated a Laue back-reflection pattern.

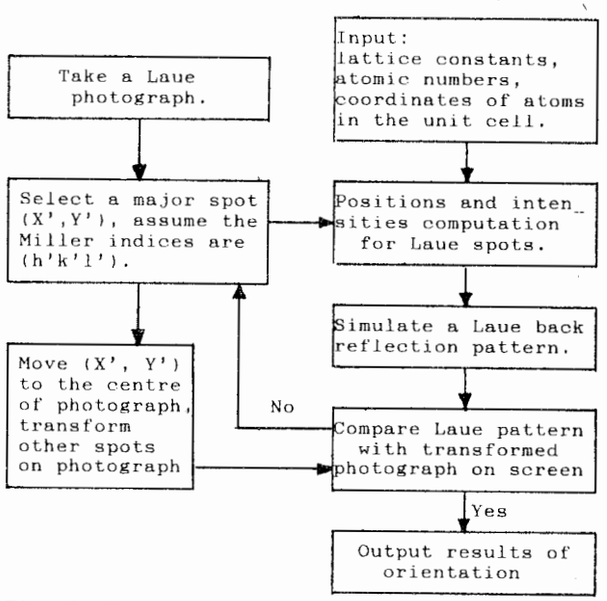

Fig. 1 Schematic flow chart of CACO.

The relative intensity of each reflection is calculated by using the structure factor F, the Lorentz factor L, the absorption correction,the geometric factor and Kramer's formula (Preuss, Laue Atlas, John Wiley, 1973).

4. Compare the Laue back-reflection pattern with the transformed photograph on screen.
5. If the simulated Laue pattern coincides with photograph,the assumption in step 2 is correct, the crystal orientation is finished; otherwise the predefinition is wrong, go to step 2.

The computer program of CACO is written in BASICA and designed to run on IBM-PC/AT or compatible computers. It can be used not only for orienting single crystals, but also for plotting Laue back-reflection diagrams and stereographic projections of any crystal structure. By changing some details, CACO can also be applied to transmission Laue method.

PS-01.03.09 A HIGHLY PARALLEL IMAGING GAS COUNTER FOR SYNCHROTRON RADIATION DIFFRACTION

By R.A. Lewis*, N.S. Fore, C. Hall, W. Helsby, A. Jones, B.Parker, I. Sumner, J.S. Worgan. S.E.R.C. Daresbury Laboratory, Warrington, WA4 4AD, England

Multiwire Gas Proportional Counters (MWPCs) currently in use for synchrotron radiation diffraction offer unrivalled dynamic range and detection efficiency. Unfortunately they have until recently been somewhat limited in count rate performance and suffer from parallax problems at high angles of incidence. We report here on the design of a new fast area detection system currently under construction for the Daresbury SRS. It utilises a highly parallel data acquisition system in order to achieve photon counting rates in excess of 10^6 counts per second, coupled to a pressurised proportional counter to reduce parallax. The detector will be 200mm x 200mm and the system will have a real spatial resolution of ~ 200μm.

Recent test results from functioning parts of the system are shown and an evaluation of the merits of using Microgap versus conventional detector designs are reported.

PS-01.03.10 AN IMAGING-PLATE (IP) AREA DETECTOR SYSTEM DEVELOPED FOR HIGH SPEED DATA COLLECTION FROM LARGE-UNIT CELL CRYSTALS USING A ROTATING ANODE GENERATOR.

By M. Sato*, N. Tanaka†, Y. Katsube and T. Higashi¶, Inst. for Protein Research, Osaka University, †Tokyo Inst. of Technology, ¶Rigaku Corporation, Japan.

The imaging plate area detector system (R-AXIS IIC) using a rotating anode X-ray generator was developed for high-speed data-collection from large-unit-cell crystals (Sato *et al.*, J. Appl. Cryst., 1992, 25, 348-357) It is a fully automatic data aquisition system without manual intervention. The diffraction geometry is based on an Arndt-Wonacott oscillation camera, except that the crystal is rotated around a vertical spindle axis. A double-focusing X-ray optics that uses Ni coated mirrors polished like an arc is employed to avoid unfavorable curvatures arising from conventional mechanisms to bend flat mirrors. It is quite suitable for collecting data from large-unit-crystals. Two kinds of software packages are provided: one controls

the system mechanically, takes still or oscillation frames, the other processes the frames to obtain a set of reflection data. The orientation matrix of the crystal is determined by Higashi's auto indexing algorism. The processing of oscillation images is based on Wonacott's strategy for the oscillation-film method and estimates integrated intensity by Rossmann's five quadrant profile fitting algorism. Scale and temperature factors are refined by the method of Fox and Holmes. The quality of diffraction data was evaluated as follows; R-merge values being 0.04 ~ 0.05 range and data reproducibility better than that of a four-circle diffractometer, isomorphous and anomalous difference Patterson maps showing significant peaks corresponding to heavy-atom vectors, electron density maps being of high quality enough to asign amino-acids unambiguously, data collection rate being fast enough to collect a full set of diffraction data from one protein crystal.

PS-01.03.11 THE FIRST EXPERIMENTS WITH A HIGH POWER WIGGLER AT THE THIRD GENERATION EUROPEAN SYNCHROTRON RADIATION FACILITY
Å. Kvick, European Synchrotron Radiation Facility, BP 220, F-38043 Grenoble Cedex, France

Twelve European countries have jointly built a 6 GeV third generation synchrotron radiation facility in Grenoble, France. The storage ring was successfully commissioned during 1992 and is now running routinely with 100 mA of electrons stored with lifetimes well in excess of 10 hours. The emittance is in close agreement with the predictions.

The installation of the first three beamlines was started in June 1992, and first beams in the experimental hutches were obtained in November 1992. This presentation will concentrate on beamline 2 also referred to as "The Materials Science" beamline, which is based on a 1.24 T multipole wiggler with 12 periods of 125 mm length. This wiggler is the highest power and highest flux insertion device presently planned at the ESRF. The total generated power is 5.6 kW and the characteristic energy at a 20 mm gap is E_c=30 keV (0.4 Å). The wiggler thus gives total integrated fluxes of close to 10^{15} photons/sec/0.1% over a wide energy range (10^{14} for instance at 80 keV).

The beamline was commissioned to full power operation during the brief November and December operational periods, and early experiments both with white beam and monochromatic beam have been performed since December 1992. To date experiments including high temperature (over 1900° C) phase transitions of perovskites, high pressure (180 kbars) phase transitions of Ge, complete data collections from proteins at short wavelengths (0.45 Å) using both imaging plates and imaging intensifier detectors, powder diffraction patterns of molecular sieves using imaging plates have been performed, and extensive further test are planned for the spring and summer periods. The presentation will concentrate on the results from the crystallographic experiments performed with this new source of synchrotron radiation.

PS-01.03.12 INEXPENSIVE UPGRADE OF A NICOLET P3M DIFFRACTOMETER. C. Svensson and B. Sommarin*, Inorganic Chemistry 2, Chemical Center, University of Lund, PO Box 124, S-221 00 LUND, Sweden.

The Data General Nova control computer of our Nicolet P3m single-crystal four-circle diffractometer has been out of order for some time. Because the motor controllers and drivers are integrated with the computer, the whole drive chain had to be renewed. We have connected the goniostat to a PC containing a commercial ISA-bus motion controller board and added an external unit with power supply and four servo amplifiers. To minimize the amount of work on the goniostat itself, the original DC motors have been retained, but the angle resolvers have been replaced by optical encoders using the original resolver mounts. Limit and zero point sensors have been added. The step size is 0.001° for all axes with a positioning precision of a quarter of a step. The setup of the servo system is facilitated by the tuning software supplied by the motion controller manufacturer.

The comprehensive crystallographic control software that is used for the upgraded instrument is the same as was developed for our Huber four-circle diffractometer (Svensson and Ståhl, J. Appl. Crystallogr., 1993). Only minor software modifications were necessary for the modified P3m. A new set of routines was written for the microprocessor-based motion controller board. These are downloaded as needed. Once the controller has received a command it is self sufficient and will run independent from the CPU and memory of the PC until completion. Several options on the general purpose board enhance the performance of the diffractometer, eg. the electronic gearing, the automatic S-curve profiling during acceleration and deceleration, the high speed position capture, and the extensive error handling. The controller also has analog-to-digital converter inputs that can, for example, be used for logging the temperature during a low-temperature experiment.

PS-01.03.13 A LABORATORY DISPERSIVE EXAFS SPECTROMETER. By J. Galy, P. Baules, J. Jaud, P. Lecante, A. Mosset,* CEMES-LOE / CNRS, 29 Rue J. Marvig, BP 4347, F31055 Toulouse Cedex, France.

The study of short range order in non-crystalline or poorly crystallized materials usually involves LAXS (Large Angle X-ray Scattering) and/or EXAFS (Extended X-ray Absorption Fine Structure) measurements. Synchrotron radiation emitted by large storage rings constitutes the best source currently available for EXAFS experiments. However, the practical interest of a laboratory facility promoted the development of various devices making use of the weak Bremstrahlung spectrum produced by sealed-off or rotating anode X-ray tubes. Most of existing in-lab spectrometers use Johanson or Johann type bent monochromators and work in sequential mode.

An alternative technique makes use of the divergent character of the beam produced by X-ray tubes. X-rays falling on a single crystal settled in transmission or reflection geometry with different incidence angles are diffracted for Bragg related different energies. All intensities can then be simultaneously recorded using a linear position sensitive detector. Mechanical motion suppression during data collection decreases measurement times and also reduces potential drift and background, allowing long exposures in the case of low intensity X-ray sources or diluted samples.

In this paper, we report the principle and the construction of a laboratory EXAFS spectrometer optimized for transmission dispersive mode. Factors determining energy range and resolution are detailed. An empirical energy calibration procedure is proposed. EXAFS spectra obtained for copper metal foil, molecular complex copper acetate, germanium oxide and zirconium oxychloride compare favourably with data obtained using conventionnal synchrotron facility.

PS-01.03.14 HX-1B FULLY MICROCOMPUTERIZED FOUR CIRCLES SINGLE CRYSTAL DIFFRACTOMETER CONTROL SYSTEM AND AUTOMATED CRYSTAL STRUCTURE ANALYSIS SYSTEM. By Shen Jinchuan*, Jin Xing, Yang Daihua and Liang Jun, Test Centre, China University of Geosciences, Wuhan 430074, P.R. China.

On the basis of an obsolete Picker Single Crystal Diffractometer