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

The system, mechanically, is still in oscillation frame, the other procedures the frame to obtain a set of reflection data. The orientation matrix of the crystal is determined by Higashi's auto-indexing algorithm. The processing of oscillation images is based on Hooke's strategy for the oscillation method and estimates integrated intensity by Rossmann's two-quadrant profile fitting algorithm. 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 X-ray Patterson maps showing significant peaks corresponding to heavy-atom vectors, electron density maps being of high quality even to assign 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

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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 electron stored with lifetimes well in excess of 30 hours. The commissioning 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 hutch were obtained in November 1992. This presentation will concentrate on beamline 1 also referred to in the Materials Science beamline, which is based on a 1.24 T multipole wigglers with 12 periods of 125 mm length. This wiggler is the highest power and highest flux reaction device presently planned at the ILSRF. The total generated power is 40.2 kW and the characteristic energy at a 30° gap is E = 170 eV (0.4 Å). The wiggler thus gives total integrated fluxes of close to 1015 photons/sec/Å2/1° over a wide energy range (102 for instance at 80 keV).

The beamline was commissioned to full power operation during the brief November and December operation periods, and early experiments both with white beam and monochromatic beam have been performed since December 1992. To date experiments including high temperature (over 1700° C) phase transitions of perovskites, high pressure (100 GPa) phase transitions of Ge, complete data collections from proteins at short wavelengths (0.4 Å) using both imaging plates and imaging intensifier detectors, powder diffraction patterns of molecular sieves using imaging plates have been performed, and extensive further tests 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

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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 drives are integrated with the computer, the whole drive chain had to be renewed. We have connected the generator 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 ammount of work on the gantry 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 0.01° 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 Stål, 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 aspects on the general purpose board enhance the performance of the diffractometer, e.g. the electronic gearing, the automatic S-curve profiling during acceleration and deceleration, the high speed position capture, and the excessive 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 DISPERSE S EXAFS SPECTROMETER

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The study of short range order in non-crystalline or poorly crystallized materials usually involves EXAFS (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 laboratory facility promoted the development of various devices making use of the weak Bremsstrahlung spectrum produced by sealed-off or rotating anode X-ray tubes. Most of existing in lab spectrometers use Johnson's or Johanna 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. The 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 suitable 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 stearate, germanium oxide and zirconium oxychloride compare favourably with data obtained using conventional synchrotron facility.

PS-01.03.14 HX–13 FULLY MICROCOMPUTERIZED FOUR CIRCLES SINGLE CRYSTAL DIFFRACTOMETER CONTROL SYSTEM AND AUTOMATED CRYSTAL STRUCTURE ANALYSIS SYSTEM. By Shen Jinchun*, Jin Xing, Ying Duhua and Liang Jue, Tens Centre, China University of Geosciences, Wuhan 430074, P.R. China.

On the basis of an obsolete Picker Single Crystal Diffractionmeter