

**PS01.02.13 INSTRUMENTATION FOR X-RAY FIBRE DIFFRACTION UNDER INDUSTRIAL PROCESSING CONDITIONS.** Arumugam Mahendrasingam, Physics Department, Keele University, Staffordshire, ST5 5BG, UK

A purpose-designed x-ray fibre diffraction camera has been built which when used in conjunction with three synchronised CCD cameras allows the simultaneous recording of wide angle x-ray scattering (WAXS), small angle scattering (SAXS) and strain data. X-ray data recording is by a Photonics Science CCD detector linked to a Synoptic i860 framegrabber. Full two-dimensional diffraction patterns can be recorded with exposure times as short as 40 milliseconds with sufficient on-board memory in the framegrabber to allow up to 128 successive frames each of 512 x 512 pixels to be recorded before diffraction images are downloaded to a dedicated PC. Strain rate and draw ratio at the point in the specimen from which the diffraction data is recorded is calculated from changes in the position of reference lines on the specimen. Programmes have been developed to display and analyse the x-ray diffraction data in the x-windows environment under the Linux operating system. This system allows the variation in orientation and crystallinity in polymer materials to be studied under industrial processing conditions, i.e. strain rates up to 150,000% per minute at temperatures up to 350°C. The system is designed to be portable and has been used on in-house rotating anode generators as well as at the Daresbury Laboratory Synchrotron Radiation Source and the European Synchrotron Radiation Facility, Grenoble. The capability of the system will be illustrated by studies on a variety of organic polymer materials and artefacts fabricated from them.

**PS01.02.14 XANES REGISTRATION BY ELECTRON BEAM POSITION SCANNING FOR TIME-RESOLVED EXPERIMENT.** Sergei G. Nikitenko, \*Boris P. Tolochko, \*\*Alexander N. Aleshchev, \*\*Gennady N. Kulipanov, \*\*\*Svyatoslav I. Mishnev. Institute of Catalysis, Novosibirsk-90, Russia; \*Institute of Solid State Chemistry, Novosibirsk-128, Russia; \*\*Institute of Nuclear Physics, Novosibirsk-90, Russia

We have designed and realized at VEPP-3 new method for XANES registration. Traditionally, the monochromator rotation is used for the energy scanning. In new method the position of the monochromator is fixed, but the position of the electron beam changes by magnetic field. As a result the angle SR-beam/monochromator changes and changes the energy of the monochromatic beam.

We received test-XANES spectra of Ag by electron beam position scanning and then use this method for investigation of the fast Ag reduction from metal-organic compounds.

Depending on the type of the monochromator used and energy interval scanned, XAFS spectra may now be measured in a matter of 6-20 s. Traditional QXAFS has natural limit, which depends from the mass of the crystal of the monochromator, holder and translation stage. The mass of the electrons in the bunch is very small and there is no limit for fast scanning.

The using undulator as a SR source in this method can improve the time resolution of this method in several orders.

**PS01.02.15 VACUUM CHAMBER FOR SYNCHROTRON POWDER DIFFRACTION.** B. Palosz, S. Gierlotka, S. Stel'makh, S. Doyle† & T. Wroblewski‡. High Pressure Research Center "UNIPRESS", Warsaw, POLAND; † HASYLAB, DESY, Hamburg, GERMANY

Air scattering is a primary concern during experiments where weak and/or diffuse intensities are measured. We designed, manufactured and tested a chamber that allows for measurements of samples under vacuum at B2 powder diffractometer at HASYLAB, Hamburg, Germany. The chamber is a flat half-cylinder with 300 mm radius. Kapton window 15 mm

wide covers 2θ range from -5 to 130 deg. The chamber is connected via a flexible bellow to the beam-leading tubes that reach the exit window of the monochromator tank where another kapton window is placed. Therefore the beam-definition slit, the beam monitor and the anti-scatter slit are under vacuum. The total length of the vacuum beam path is roughly 1600 mm. The sample is mounted on a goniometer head inside the chamber with all necessary degrees of freedom. Tests were performed with a scintillation detector and the wavelength of 1.158 Å. The beam stop was a strip of lead attached to the chamber right behind the kapton window. It is positioned precisely by rotating the whole chamber mounted on the diffractometer circle. In standard setup the measurements start from 0.35 deg. 2θ; lower angles can also be reached. For a short wavelength, 1.158 Å, there was only little gain in the primary beam intensity. But the instrumental background at small and intermediate angles was reduced by more than 2 orders of magnitude. Test measurements were performed on nanometric diamond and SiC samples. The setup allowed for simultaneous recording of small- and wide-angle data. The SAXS curve extended over 5 decades and the diffuse intensity in the intermediate angles was accurately determined. This chamber equipped with curved Image Plate will be a very effective tool for quantitative measurements of the intensity profiles of conventional polycrystals, nanocrystalline and non-crystalline materials.

**PS01.02.16 LAUE DATA REFINEMENT OF THE CRYSTAL STRUCTURE OF STANFIELDITE, DATA COLLECTED AT CHESS USING UNDULATOR RADIATION WITH IMAGE PLATES IN A CYLINDRICAL FILM CASSETTE**

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X-ray Laue diffraction data were obtained for Stanfieldite using an exposure time of 0.05 seconds per frame for 9 frames. The data were processed using LaueView (Ren, Moffat, J. Appl. Cryst. (1995) 28,461).

Sample Source: stony-iron meteorite

$\text{Ca}_{4.05}\text{Mg}_{4.55}\text{Fe}_{0.32}\text{Mn}_{0.05}\text{P}_6\text{O}_{24}$

Space Group C2/c

Unit Cell: 22.813(3) 9.993(1) 17.090(1) 99.96(1)

Crystal Size 0.06 x 0.1 x 0.2 mm<sup>3</sup>

	Monochromatic	Laue
X-ray wavelength	0.71069	0.7 - 3.0
exposure time	567360 sec (~6.6 days)	0.45 sec
Unique diffractions	6928	8475
R	0.047	0.1841
Rw	0.041	0.1650
Parameters varied	359	359

Although the R values for the Laue refinement remained high, the results are in good agreement with the monochromatic data set. See below for a partial listing of atom parameters (1st line monochromatic, 2nd Laue):

Atom	x	y	z	Ueq
Ca5	0.06597(2)	0.10401(6)	0.58830(3)	0.0155(2)
Ca5	0.06608(8)	0.1041(2)	0.58843(14)	0.0187(6)
Mg5	0.42063(3)	0.09020(7)	0.55203(4)	0.0089(2)
Mg5	0.42062(10)	0.0906(2)	0.5522(2)	0.0107(8)
P1	0.18317(2)	0.11846(5)	0.10024(3)	0.00689(13)
P1	0.18335(7)	0.11856(15)	0.10017(13)	0.0099(5)
O1	0.24338(6)	0.0845(2)	0.15538(9)	0.0095(4)
O1	0.2435(2)	0.0841(5)	0.1550(4)	0.0138(15)