A major objective for the single crystal diffraction instrument currently under construction at the Spallation Neutron Source (SNS), ORNL, is to make extensive use of beam transport and focusing optics. This time-of-flight Laue diffractometer will implement a super mirror beam guide following the trace of a parabolic curve in a piecewise approximation [1]. In this context micro-focusing optics under development for neutron scattering applications are also being reviewed and recently collected data from prototypical assemblies and setups are being presented.

[1] Stoica A.D., Wang X.-L., Lee W.-T., Richardson J.W., in *Advances in Computational Methods for X-Ray and Neutron Optics, Denver,* 2004, Proceedings of SPIE Vol. **5536**, p. 86.

Keywords: single crystal neutron diffraction, focusing optics, neutron instrumentation

P.01.09.1

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Single-Crystal Neutron Diffraction on Sigma Complexes: Recent Results from IPNS

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For a number of years we have been employing single-crystal neutron diffraction to investigate structures of sigma complexes of transition metals. Sigma complexes are of special interest because they are ubiquitous intermediates in metal-catalyzed reactions including hydrogenations, activation and functionalization of hydrocarbons, and hydroborations. Here we will report on some recent results obtained using the SCD instrument at Argonne's Intense Pulsed Neutron Source, which has been upgraded with two new position-sensitive Anger detectors to achieve increased data collection efficiency. In the future, we hope to be able to dramatically extend these studies at the Spallation Neutron Source (SNS) using the single-crystal diffractometer (Topaz) that is currently under development there. *Acknowledgement.* This work was supported by the U. S. Department of Energy, Office of Basic Energy Sciences, under Contract W-31-109-ENG-38.

Keywords: neutron crystallography, organometallic complexes, single-crystal diffraction

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A New Area Detector for Ultra-fast X-ray Diffraction Analysis

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A state-of-art semiconductor technology based area X-ray detector, namely D/teX-25, has recently been developed. This detector has ultra high-sensitivity and can achieve ultra high-speed X-ray diffraction (XRD) measurement up to a maximum speed of a pattern of 160º2θ in one minute or 90º2θ in about 30 seconds, which is more than 30 times faster than a conventional speed of 5º2θ per minute with a scintillation or a proportional counter. In addition to high-speed data acquisition, the D/teX-25 can provide X-ray diffraction analysis with areal resolution for the study of sample uniformity and the possible presence of large or aggregated particles in a specimen. Thus the D/teX-25 detector is useful for dynamic *in-situ* studies of various materials. Some examples of fast and/or two dimensional XRD measurements with a D/teX-25 detector will be given.

Keywords: XRD, area detector, high-speed

P.01.10.2

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Obtaining Accurate Lattice Parameters from Debye-Scherrer Image Plate Data

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Image plates (IPs) allow high-resolution, high-dynamic range registration of multiple datasets on a single IP. When refining accurate lattice parameters, independent determination of strongly correlated parameters, usually displacement and zero offset, is essential. We have investigated the use of embedded radioactive fiducial markers to absolutely calibrate the angular scale on each IP and thereby eliminate the need to refine zero offset.

We found that a random rotation of up to +/- 0.3 degrees is introduced in our BAS2000 scanner during loading of the IP. The consequent systematic variation in refined lattice parameter for multiple datasets on a single image plate seriously complicates diffraction-based thermometry. Typical variation in refined lattice parameter for data collected under identical conditions at each extremity of the IP was found to be 0.02% for uncorrected data, and 0.003% when rotation was taken into account. Thermal expansion is normally of the order of 0.005% per degree C. The effect of IP rotation is reduced when datasets from multiple IPs are refined simultaneously.

The fiducial markers also enabled detection of occasional random "skips" in IP position during scanning.

Keywords: lattice parameter refinement, imaging plates, thermal expansion

P.01.10.3

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Data Collection System for Protein Crystallography using CMOS Image Sensor

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CCD detectors are commonly used for protein crystallography at synchrotron facilities because of easy operation and high readout speed. But, the fine-slice oscillation method using highly intense X-ray of third generation synchrotron requires higher frame rate, since a large amount of diffraction images must be recorded with an exposure time of less than 1 second. Therefore, development of new detectors having higher frame rate is required. A detector utilizing a CMOS image sensor[1] is one of the promising candidates for this purpose.

We developped a data collection system for protein crystallography using a CMOS detector Shad-o-Box 4K from Rad-icon Imaging corp. It consists of 8 active-pixel CMOS sensors tiled in a 2 x 4 matrix and contains a total of 2000 x 2048 pixels of photodiodes with 48 μm spacing. Maximum frame rate of 2.7 fps is available. Images are captured into PC through a frame grabber board PXD 1000 (Imagination corp.). Server software running on Windows XP was developed using PXD 1000 frame grabber library, so that it can be controlled by client software BSS (Beamline Scheduling Software)[2], which is SPring-8 standard data collection software for protein crystallography.

We are now studying the performance of the new data collection system to use CMOS detectors as an alternative to CCD detectors.

[1] Yagi N., et al., *J. Synchrotron Rad.*, 2004, **11**, 347-352. [2] Ueno G., et al., *J. Appl. Cryst.*, 2004, **37**, 867-873.

Keywords: detector development, protein crystallography, CMOS detectors

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CMOS Flatpanel Detectors for SAXS/WAXS Experiments

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CMOS flatpanel detectors have been commercially available for some years. We tested these in several different synchrotron radiation applications [1,2], including X-ray diffraction and scattering experiments. Even with a passive-pixel device, which has a higher noise level, an R-merge value of 6% was obtained in the processing of diffraction from a lysozyme crystal [2].

In small-angle scattering experiments, the detector may eventually replace the CCD-based detectors that are currently used in combination with synchrotron radiation. Since the CMOS detector is very compact in size, it is considered especially useful as a wide-angle detector in small-angle diffraction experiments. Not only it is possible to place the detector very close to the specimen, it is also feasible to