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

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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

P.01.10.4

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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 align the detector so that one edge of the sensor lies very close to the beam. A special detector was manufactured by Rad-icon Imaging Corp to pass X-rays between two detector sensors, which were separated by about 3 mm. With this detector, it is possible to record a two-dimensional wide-angle diffraction pattern while another detector at the downstream end of the camera records a small-angle diffraction pattern. An active-pixel CMOS detector from Hamamatsu Photonics is also suitable for this purpose.

[1] Yagi N., Yamamoto M., Uesugi K., Inoue K., *AIP Conference Proceeding*, 2004, **705**, 344. [2] Yagi N., Yamamoto M., Uesugi K., Inoue K., *J. Synchrotron Rad.* 2004, **11**, 347.

Keywords: detector properties, synchrotron radiation, small-angle X-ray scattering

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1- and 2D Detectors and Sample Fluorescence in XRD

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In the last decade, electronic 1- and 2-dimensional X-ray detectors have replaced scintillation detectors and X-ray films for both Single Crystal, and Powder Diffraction. For many applications, these detectors bring orders of magnitude increase in data collection efficiency, and as a result of that, they allow us to increase productivity and/or collect data with a higher accuracy within a reasonable time frame.

A consequence of these more ‘open’ detection systems is that they are more susceptible to scattered radiation, and also to photons with a wavelength differing from the chosen characteristic radiation, such as K-Beta radiation and sample fluorescence. This is especially visible in powder XRD as the traditional Bragg-Brentano diffractometer geometry includes a focusing diffracted beam monochromator, which takes out a lot of unwanted radiation.

For high-speed X-ray detectors, a focusing diffracted beam monochromator does not exist. This does not mean, however, that it is not possible to record diffractograms with a good peak-to-background ratio. Especially with sample fluorescence, there are a lot of options for obtaining a good result and this contribution is aimed to help selecting the best one. Measurements were analyzed which were carried out on samples of the first row of transition metals, ranging from Ti to Ga as a function of the following variables: tube anode material, use of incident or diffracted beam monochromators, use of incident or diffracted beam beta filters, Pulse Height Discrimination settings, and generator settings.

Keywords: X-ray detectors, X-ray powder diffraction techniques, experimental design

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Use of a Single Crystal Diffractometer and CCD Area Detector for Phase Identification

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CCD-based single crystal X-ray diffractometers are widely used in the fields of chemistry, materials science, biology and mineralogy for crystal structure determination. However, CCD detectors have not yet been widely utilized in the field of powder diffraction.

A new Phase Identification option has now been integrated into the Bruker APEX2 software suite, so that the same hardware and software may be used for both structure determination and powder diffraction measurements. This module will be offered as an optional add-on feature for the small molecule single crystal instruments using the APEX2 (Version 2.0) software suite. For more specialized applications, data may also be exported to the Bruker DIFFRAC[plus] (EVA & TOPAS) programs.

The powder diffraction option is intended to supplement the primary use of a single-crystal diffractometer to verify that the sample is a single phase or polymorph and that the analyzed single crystal specimen is representative of the bulk sample.

Various examples of powder diffraction patterns collected on Bruker Kappa APEX II and SMART APEX II instruments equipped with Cu- or Mo-wavelength X-ray sources will be presented.

Keywords: powder diffraction, CCD detectors, software

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IP Slant-incidence Correction for Accurate Structure Factor Measurements

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Slant incident X-rays on IP give higher intensity than normal incident ones. The correction for the effect was first proposed by considering incomplete absorption of X-rays in the phosphor layer of IP[1]. We proposed a correction factor cosí taking into account the absorption of emitted luminescence by the phosphor layer[2]. However they are not good enough at higher slant angles. This inhibits us to develop highly accurate IP devices, although IP has very high potential for accurate structure factor measurements.

The cosv formalism was improved by carefully evaluating the accessible area of emitted light by the optical system of the IP readout system, BAS2500(Fuji Film) and applied it to 4f-electron density measurement of CeB_6 by VCIP method. The ratio of the difference of observed and calculated intensities to calculated one were plotted against í for all the observed reflections, exhibiting excellent coincidence over the observed írange from 0 to 55°. Actually R factors without correction, with correction employing cosí formalism and using the new method were 4.5, 2.9 and 1.9%, respectively. 4f-electron deformation density in CeB_6 appeared only after the new correction, which is similar to the one by four-circle diffractometer[3]. The method can be easily extended to the other IP readout systems.

[1] Zaleski J., Coppens P., *J. Appl. Cryst.*, 1998, **31**, 302. [2] Zhurova E. A.,Zhurov V.V., Tanaka K., *Acta Cryst.* 1999, B**55**, 917. [3] Tanaka K., Onuki Y., *Acta Cryst.*, B**58**, 423.

Keywords: image plate, slant-incidence, 4f-electron density

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XPAD: A Pixel Detector for Material Sciences

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Currently available 2D detectors do not make full use of the high flux and high brilliance of third generation synchrotron sources. For this reason numerous experiments are still performed using slits and photomultipliers that allow only point detection. At the present time, the 2D detectors in most common use are CCD cameras with indirect photon detection.

The XPAD photon counting detector has been developed for materials science and small angle scattering experiments similar to those performed on the CRG-D2AM beamline at ESRF. At the time, its prototype is built of 8 modules of 8 chips for a total area of about 6.8x6.8 mm^2 and 200x192 pixels.

Recent results of powder diffraction of CaSrX zeolite [1] have prooved that such 2D dete ctors present a new opportunity to improve the quality of our measurements. SAXS results will also be presented and compared to CCD ones.

[1] Basolo S., Berar J.-F., Boudet N., Breugnon P., Caillot B., Clemens J.-C., Delpierre P., Dinkespiler B., Koudobine I., Meessen Ch., Menouni M., Mouget Ch., Pangaud P., Potheau R. , Vigeolas E., accepted in IEEE Trans. Nucl. Sci., conference IEEE-2004, Rome.

Keywords: detector development, synchrotron radiation, materials science