structures determination and for application of the Rietveld method in quantitative phase analysis. The utilization of this instrument is open for the brazilian and latin-american scientific and technological communities.

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Keywords: neutron diffractometer, PSD detector, focusing monochromator

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The new Single Crystal Diffractometer HEiDi at the FRM-II and its Applications

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HEiDi, one of the new single crystal diffractometers of the research neutron source FRM-II, was designed to cover a wide area of scientific applications in crystal structure analysis. It uses the high flux of fast neutrons with short wavelengths from the hot source of the FRM-II. The enlargement of the visible reciprocal space (=Q-space) allows very accurate determinations of nuclear positions in single crystals as well as more detailed quantitative informations about mean square displacements and vacancies which is of interest in reference to static or dynamic disorder effects and phase transitions. The Q-dependences of the magnetic and the nuclear cross sections of neutrons are quite different. This can be used to determine the magnetic and the nuclear order in a crystal separately. Other advantages of shorter neutron wavelengths (1.4 Å down to 0.3 Å) are the significant reduction of absorption effects in compounds with highly absorbing elements (e.g. Sm, Gd) and the reduction of extinction effects.

During the nuclear commissioning of the FRM-II in 2004 started the adjustment and characterization of HEiDi with neutron radiation. First experimental results are quite promising, e.g. an excellent resolution function (<0.1° at min.) or a perfect alignment between the calculated and the measured gain factor of 2.5 of the monochromator focussing unit. Further experimental results from the instrument and typical applications like structural phase transitions, local disorder (H bonds in RDP) or magnetism will be presented on the conference.

Keywords: neutron and X-ray diffractometry, single crystal structure analysis, neutron instrumentation

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The Italian Neutron Experimental Station (INES) at ISIS: Status and Development

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The INES project concerns the realization of a multipurpose experimental station, built by CNR at the ISIS pulsed neutron source (Rutherford Appleton Laboratory, UK). This instrument is mainly intended to operate as test and training facility for the Italian neutron-scattering community. The experimental station is equipped with a multipurpose time-of-flight neutron diffractometer, presently under commissioning. This is located downstream a water moderator of the neutron source, with an excellent time-resolution. In the present configuration the INES diffractometer contains a highly-efficient large detector area covering a range of about 170^{o} on the horizontal plane. Moreover it offers a large sample volume (about 0.25 m^3), allowing the study of almost any kind of object, including bulky archaeological artifacts. The possibility to separately analyze each single detector makes texture analysis also possible. The opportunity to operate experiments in particular thermodynamic conditions (i.e. high pressure, high and low temperatures) is also under investigation.

Keywords: neutron istrumentation, texture analysis, archaeometry

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Design of a High Resolution Macromolecular Neutron Diffractometer (MaNDi) for Structural Biology Research at the SNS

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With the advent of third-generation synchrotron X-ray sources, it was envisioned that ultra-high resolution macromolecular crystallography (UHRXMC) at resolutions of 0.5 Å to 1.0 Å would provide detailed information on the positions of critical hydrogen atoms within the active sites of enzymes. To date, in about 82 structures in the PDB in this resolution range, significant numbers of hydrogen atoms including those in the active sites could not be identified. Furthermore, only about 0.5% of all macromolecular structures in the PDB are amenable to UHRXMC and hence other complementary techniques are needed for the identification of critical hydrogen atoms involved in the catalytic mechanisms in a majority of enzyme systems.

Neutron Macromolecular Crystallography (NMC) has been shown to provide accurate proton positions, protonation states and hydration states, as well as hydrogen/deuterium exchange, in macromolecular crystals even at moderate 2 Å to 2.5 Å resolution. One major bottleneck that severely constrains the productivity of NMC is the limited flux at the current sources and the requirement of large crystals. The advent of the Spallation Neutron Source (SNS), with over an order of magnitude increase in neutron flux, the advances in neutron optics and detectors, as well as advances in structure genomics and deuteration. provide an exciting opportunity to push the NMC field to new horizons. Hence we propose to develop a dedicated world-class high resolution time-of-flight single crystal macromolecular neutron diffractometer (MaNDi) for structural biology research at the SNS. MaNDi has been designed to be able to collect a full hemisphere of Bragg data with a resolution of 1.5 to 2 Å on a crystal with a lattice constant up to 150 Å in 1 to 7 days. The higher throughput and resolution are accomplished by the use of a wide wavelength bandwidth of cold neutrons (1.8 Å < λ < 4.5 Å) sorted into a large number of high resolution wavelength channels by time-of-flight and by an array of high resolution position-sensitive area detectors covering a large solid angle. We envision that the unprecedented high data rates and resolution with MaNDi will open up new avenues and greatly advance the field of structural biology, enzymology and protein dynamics.

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Keywords: neutron macromolecular crystallography, time-of-flight single crystal diffractometer, structural biology

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Advances in Neutron Single Crystal Diffraction towards a Smaller Sample Sizes

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Single crystal diffraction has been used as a tool for structure analysis since the discovery of neutron scattering. Complementary to X-ray radiation neutron radiation is especially useful to locate 'light' elements like hydrogen next to 'heavy' elements like metals. Furthermore, neutrons are much "gentler" to organic crystals. A major obstacle for neutron diffraction is the moderate flux and therefore the significantly larger single crystal sizes and longer data collection times needed for a decent data set.

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

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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