P.01.11.2
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Metrological Assurance of the Substance and Materials Investigations by Diffraction Methods
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Volume and reliability of information about the properties of crystalline substances and materials are frequently determined by the optimum experiment planning and application of the corresponding equipment. However, solving of reverse type problem on the obtaining the characteristics of crystals from the diffraction pattern is determined by the level of the development of data processing methods and by the quality of the chosen starting model. The latter component is connected to some extent with intuition and researcher confidence in the admissibility of the model used.

Crystals Metrology Dept. (CMD) develops metrological assurance for the diffraction measuring instruments. For practical Crystals Metrology this assurance includes the system of the Certified Standard Reference Materials (SRM) diffraction properties of materials, Procedures of Measurements, Databases, Sequence of test steps and other normative documents for the tests according to declared functions (designations) of diffraction equipment (for Type Approval and conformity tests). CMD organizes and processes the data of interlaboratory experiments of round-robin type, carries out its own experiments of high and the highest accuracy for some poly- and single-crystals key materials of modern technologies, develops the methods for investigation and characterization of new complex substances, including certification of medications and determination of resources of materials and products made of these materials. The task of establishment of reproducible Mass Unit (both as the characteristic of a Substance Quantity and the characteristic of its inertness measure - gravitational constituent - namely the Kilogram Unit on the basis of high-clean certified Silicon) remains urgent among CMD basic tasks of fundamental Diffraction Crystals Metrology the same as the task of the interrelation of the basic units of SI.
Keywords: materials metrology, data accuracy, standard reference samples

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The EU BIOXHIT Standard Test Crystal
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The standard test crystal is intended to provide an automated, fast and robust procedure for identifying potential problems in the complex hardware and software infrastructure of modern protein crystallographic (PX) synchrotron beamlines.

A quick test dataset should be collected whenever changes have been made to the hardware or problems are suspected. Indeed, for highly automated pipelines the first crystal in the dewar could be a test crystal in case the robot drops it! The test crystal should have high symmetry so that a short rotation about a single axis suffices to collect redundant data, the crystals should be easy to obtain and freeze reproducibly giving a small mosaic spread. The cubic form of insulin fulfils all these conditions and has been used in our tests so far, however we are also looking for possible inorganic test crystals for Se-MAD beamlines.

The diagnostics should be independent of the data integration software employed, so as a first step we have compared the processing of cubic insulin data using the widely used programs XDS, MOSFLM/SCALA and HKL2000. We will present our experiences with test crystal data collected on a number of the participating beamlines in the BIOXHIT consortium.

We are grateful to the EU for support (LHSG-CT-2003-503420) and to the other BIOXHIT partners for the fruitful collaboration.
Keywords: synchrotron structural biology research, diagnostics, test crystal

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Strain Analysis using High Energy X-ray White Beam Diffraction
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One of the principal advantages of energy-dispersive diffraction for the determination of macroscopic average lattice parameters (and hence strain) is the possibility of refinement of the large section of the diffraction pattern, leading to improved accuracy and stability of interpretation. Precise channel to energy conversion is very important in full-pattern refinement in energy-dispersive X-ray diffraction. The channel to energy conversion of most detectors is not entirely linear. This presents an obstacle to obtaining accurate quantitative data for lattice strains by pattern refinement. We present a procedure for precise energy calibration determination, and show how the new energy conversion function was used successfully to perform whole pattern fitting of energy-dispersive X-ray diffraction patterns of Ti64 samples. The strain across the Ti64 bar calculated from the fitting results was compared with the profile obtained by single wavelength X-ray diffraction utilising Laue monochromator, and showed excellent agreement.
Keywords: energy-dispersive diffraction, synchrotron radiation, titanium alloy

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Improving Data Quality – without having to grow new Crystals
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In order to obtain the best possible results for structure solution and refinement, it is imperative to collect the best quality data from a given crystal. This generally means measuring the highest possible resolution data. With recent advances in microfocus X-ray sources, such as the MicroStar, more brilliant sources are available to evaluate very small crystals in-house. Combining these sources with the latest developments in graded multilayer optics can result in excellent data being measured at home on samples that were previously only tractable at the synchrotron. Using a kappa-goniostat in combination with sophisticated data collection strategy software can ensure that a complete dataset is measured up to the diffraction limit of the crystal. The combination of high resolution, completeness and redundancy can improve the data quality significantly. In this study we will show examples of how data quality can be improved. It was possible to trace more residues in a dimeric single-stranded DNA binding protein, by collecting a dataset using the strategy program COSMO [1] in combination with the 4-circle goniometer on the X8 Proteum.

[1] COSMO, Data collection strategy program, Bruker AXS
Keywords: data collection, protein crystallography, detectors

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High Resolution Data Collection in the Home Lab
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Data collection on protein crystals to very high resolution (> 1.3Å) typically requires a trip to the synchrotron. Due to advances in optics and the introduction of micro-focus rotating anode generators, there has been a remarkable increase in brightness and flux density available in home laboratory systems. When combined with ultra-sensitive detectors, these systems provide an alternative means of

extending the diffraction limit of samples. In some cases, X-ray data extending out to atomic resolution is obtainable. Methods of data collection as well as example data sets will be presented.
Keywords: data, atomic, collection

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A Design for a New State-of-the-Art Diffraction Facility
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We present the details of a new state-of-the-art diffractometer being constructed in the crystallography laboratory in the University of Durham. The machine is designed for single crystal diffraction experiments, to explore new extremes in sample environments and combinations thereof.

The new machine, accessing areas of structural chemistry that have hitherto been unreachable in the home laboratory, will comprise: a high intensity X-ray beam to enable crystals too small for standard laboratory machines to be studied; a three stage Displex cryo-cooler which will have a temperature range of ~2-300 K, carried on a robust set of circles and a large, motorised CCD detector. The Displex will be capable of housing Diamond Anvil Cells (DACs), both fixed pressure and variable pressure designs. The Displex will be modified to create a laser injection point enabling sample irradiation at a variety of laser wavelengths, while at very low temperatures. The combination of beryllium housing for the sample environment and the large CCD detector require us to devise solutions to separate the beryllium scatter from the desired diffraction. These solutions will be discussed, including preliminary results from a software collimator, currently under development in the crystallography laboratory, University of Durham.
Keywords: machinery design, cryocrystallography, lasers

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Picosecond Lattice Dynamics Probed by Time- and Angle-resolved X-ray Diffraction
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Fast time-resolved X-ray diffraction using intense pulsed X-ray sources such as synchrotron radiations (SRs) has enabled us to take a "snapshot" of atomic arrangements in transient states produced by ultrashort pulse laser irradiation. So far, we have been developed a picosecond pump-probe system at the SPring-8 undulator beamline by synchronizing a mode-locked laser and the SR pulses [1]. By the synchronization system, the transient lattice expansion of gallium arsenide crystals by the laser irradiation has been observed, and was applied to switching of X-ray SR pulses [2].

Here, we report the acoustic phonon oscillations near the surface of a GaAs crystal observed by employing the 40 ps time-resolved X-ray diffraction, combined with angle-resolved measurement of an X-ray beam diffracted in asymmetric geometry.

The experimental results show that femtosecond laser irradiation generates the longitudinal acoustic phonon and lattice expansion along the surface normal. By decomposing the time-dependent angular distribution of diffraction into peak shift and oscillatory part, acousto-optic effect was clearly observed as out-of-phase GHz-oscillations at sidebands around the principal peak shifted due to the lattice expansion.

[1] Tanaka Y., Hara T., Kitamura H., Ishikawa T., *Rev. Sci. Instrum.*, 2000, **71**, 1268. [2] Tanaka Y., Hara T., Yamazaki H., Kitamura H., Ishikawa T., *J. Synchrotron Rad.*, 2002, **9**, 96.
Keywords: time-resolved X-ray diffraction, synchrotron X-rays, lattice dynamics

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Time-resolved Studies with Pulsed X-rays at BioCARS: Present Capabilities and Future Directions
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Static structures for many molecules are available at high resolution but the mechanism by which these molecules function and the structures of intermediate states often remain elusive. Time-resolved crystallography is a unique technique for determining the structures of intermediates and excited states in biomolecular and chemical reactions. Using the Laue X-ray diffraction technique at the high-brilliance third-generation X-ray sources (ESRF, APS, SPring-8, etc.) snap shots are taken of molecules in action with a time resolution of about 100ps, the typical duration of a single X-ray pulse at synchrotron sources. We present the status of a user facility for time-resolved studies at the BioCARS beamline 14-ID at the Advanced Photon Source. During the past years a continuously growing time-resolved user community has developed; projects under investigation include light and chemically triggered reaction mechanism. Results from most recent studies of photo-sensitive proteins will be discussed. An overview will also be given on the current efforts in enhancing the technical capabilities for time-resolved experiments at BioCARS. The technical upgrades will improve resources for complementary optical monitoring of reactions in crystals, update the laser systems, and most importantly improve the X-ray optics to enable single X-ray pulse experiments.
Keywords: time-resolved Laue diffraction, structure and function, synchrotron radiation instrumentation

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Analyzing Mosaic Domain Changes Induced by Cryo-Cooling with Digital Topography
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To better understand how cryo-cooling affects the mosaic domain structure of protein crystals a complex experiment was undertaken. Super fine φ slicing was coupled with digital topography to study diffraction at two temperatures (RT and 100K) on a single crystal, keeping the same orientation and on the same set of reflections.

For the experiment, a single lysozyme crystal was immobilized in a capillary with epoxy to eliminate slippage. The orientation was adjusted until a group of reflections were positioned to minimize Lorentz effects. The reflection group also had an arrangement that allowed them to be collected on a digital topography system in one pass. For the first part of the experiment a super fine φ slicing run was collected followed by a digital topography run at room temperature. Next the crystal was cryo-cooled in the capillary maintaining the same orientation. After cooling, a run of digital topography followed by a super fine φ slicing run was carried out on the same reflections. After processing, the sequences were analyzed to determine how the cryo-cooling had affected the mosaic domains.

The crisp mosaic domains visible in the room temperature data were shattered during cooling, the domain borders became highly irregular and some regions failed to diffract at all. Although exaggerated, the angular relationships between the major domains appeared to be conserved.
Keywords: cryo-cooling, digital topography, fine phi slicing