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THE HIGH RESOLUTION 6-CIRCLE-DIFFRACTOMETER FOR PROTEIN CRYSTAL QUALITY INVESTIGATIONS AT HASYLAB

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A 6-circle-diffractometer has been installed at the PETRA1-undulator beamline at HASYLAB. The available X-ray energy range extends from 12 keV to about 50 keV. The diffractometer is based on a Eulerian cradle and provides both horizontal and vertical diffraction planes. It has a number of point detectors, which can be combined with an analyzer crystal for high-resolution rocking curve measurements. In addition, the instrument has been equipped with a 165 mm diameter CCD detector for fast intensity data collection. This setup allows standard crystallographic data collection as well as detailed measurements of crystal mosaicity and strain. The arrangement allows different investigations without any need to remount the crystal.
For high-resolution reciprocal space mappings the incoming beam can further be conditioned by a Si-113 Bartels monochromator system. In combination with a Si-113 channel cut analyzer crystal a resolution of $\Delta d/d=5\cdot 10^{-5}$ is achieved, which is sufficient for analyzing the quality of protein crystals at high angular resolution. In first experiments the effect of radiation damage on the quality of tetragonal lysozyme crystals was investigated using a X-ray energy of 22 keV. Reciprocal space maps of the 008- and 16 16 0-reflections were therefore measured before and after exposure to an intense X-ray beam. Due to the irradiation a large increase in mosaicity has been observed. In addition an enlargement of the reciprocal lattice points in direction of the reciprocal lattice vectors has been found, indicating a variation of d-spacing within the crystal and/or particle size effects.

Keywords: PROTEIN CRYSTAL QUALITY HIGH RESOLUTION DIFFRACTOMETER RADIATION DAMAGE

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INVESTIGATION OF LATERAL HETEROSTRUCTURES OF INVERSION DOMAINS IN $LiNbO_3$, $KTiOPO_4$ and $KtiOAsO_4$

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Periodically domain-inverted (PDI) ferroelectric crystals have been of interest for optical applications for a number of years. Because the periodicity is of the order of microns, they have also proved interesting systems for study using advanced x-ray ray sources: providing a means of examining the Talbot effect, producing interesting phase-contrast images [1] and having given rise to the diffraction technique of combined “Bragg-Fresnel” (BF) imaging [2][3].
The twinning between ‘up’ and ‘down’ domains can be inferred by simulation of BF imaging results. High-resolution diffraction (HRXD) techniques were then employed to see if further structural information could be elucidated [4].
HRXD results from periods of 5μ, 9μ and 36μ were collected. In all cases remarkably clear satellite reflections corresponding to the period of the lateral grating were observed. Imaging suggested the principal cause of the satellite intensities should be phase differences in the structure factors Fhkl and F-h-k-l. Analysis shows this explanation to be inadequate, however the results do provide the opportunity of source characterisation.

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Keywords: **BRAGG FRESNEL IMAGING HIGH RESOLUTION DIFFRACTION COHERENCE LENGTH**

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ID23: A NEW BEAMLINE COMPLEX FOR MACROMOLECULAR CRYSTALLOGRAPHY AT THE ESRF

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The ESRF macromolecular crystallography resources are currently overloaded, and the situation will deteriorate as structural genomics pipeline output and industrial activity increase over the next few years. To counter this, a new beamline complex (two stations plus complete ancillary laboratories and user spaces) will be constructed on the id23 sector of the esrf.
The beamlines will be designed from the ground upwards with a high degree of automation and user friendliness in mind - building upon the experience gained from ID14 and ID29. This will include automatic alignment of optics, mounting of samples and subsequent alignment. The beamline is targeted at high-throughput crystallography with a strong emphasis on high availability and reliability of the stations. Databases will record beamline component positions as well as data collection parameters, allowing rapid beamline recovery in the event of failure and also possible remote management of the beamline and data sets.
The X-ray sources will be two canted undulators producing beams with 1.5mrad angular separation allowing two stations to operate with minimal interference. One station will be a tunable (7-20 keV) MAD-capable station and the other a fixed energy station. Available space in the storage ring will limit undulator sources to one insertion device per station. In-vacuum undulators will be difficult to install, though devices using shorter carriages may be possible in the longer term. Beamline construction is already underway and user operation is expected to be by autumn 2003 for the fully tunable station and autumn 2004 for the side station.

Keywords: **BEAMLINE, MACROMOLECULAR CRYSTALLOGRAPHY, AUTOMATION**

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RECONSTRUCTION OF DIFFUSE SCATTERING FROM CCD SYNCHROTRON DATA. EXAMPLE Nd_2GeMoO_8

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Studies of diffuse scattering using modern (commercial) CCD-detectors and synchrotron radiation sources require special data collection strategies as well as enhanced numerical data processing capabilities. A program system was developed which covers different scaling options, correction of sample external scattering, different filter options, algorithms for reciprocal space reconstruction and further numerical options for raw data processing. To enhance portability, the software consists of a series of command line based programs which are invoked from a graphical user interface. The programs were successfully applied in a study of Nd_2GeMoO_8. Compounds of the composition RGe_2MoO_8 (R = rare earth element) show significant and reversible changes of the optical absorption upon heating and a highly anisotropic magnetic behavior. The average structure ($I4_1/a$) is similar to scheelite ($CaWO_8$) with Nd occupying the Ca-positions of the scheelite structure and both Ge and Mo at the W-positions. Since laboratory studies revealed weak diffuse scattering further experiments where done at beamline F1 (Hasylab/DESY) using a CCD-detector, a wavelength of 0.071 nm and distances of up to 160 mm. Different slices in reciprocal space on a regular grid were reconstructed from the irregular spaced experimental data including corrections for refined detector- and crystal misalignments and related parameters. The reconstructed reciprocal planes show broad diffuse scattering maxima centered around positions with 0.5 a* and a significant elongation along c*, giving a coarse pattern of rod-like diffuse scattering. The distribution implies a substantial degree of chemical short-range order (no q-dependence) within the basal tetragonal plane and far smaller correlation lengths perpendicular to it.

Keywords: **SYNCHROTRON, CCD, DIFFUSE SCATTERING**