

Poster Sessions

At the companion beamline, AMX, with an intense beam of 5-100 μm suitable for investigating large complexes, the challenge of making efficient use of beam time dictates a high degree of automation. We plan to deploy a fast specimen automounter supported by a puck-loading machine working through the hutch wall. Further, the projected short data collection times dictate the time-shared use of the beam by several investigators. Already, crystallographers using the NSLS X29 undulator beam have welcomed its short beam time allocation method, and those at the similar X25 line, now equipped with a Pilatus 6M detector, experience what may evolve into (or return to) an asynchronous data collection method (collect now, analyze later).

At the SM3 beamline, emphasis will be on the acquisition of absorption, fluorescence, and Raman spectra interleaved or nearly simultaneously with X-ray diffraction measurements. Several of us have redeveloped the NSLS X26C beamline so that complementary structural information obtained by these methods now routinely provides new insights into enzymatic cycles as well as into the effects of radiation damage.

At the NYX beamline, the intellectual successor to NSLS beamline X4A, micro beams of 5-50 μm and of very high energy resolution ($\Delta E/E$ of 5×10^{-5}) will benefit the work of structural biologists focusing on challenging problems at the forefront of the field.

Additional life sciences beamlines will complement the four programs summarized above. Visit [1] for complete information.

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[1] NSLS-II: <http://www.bnl.gov/nsls2/default.asp> ; Approved beamlines: <http://www.bnl.gov/nsls2/2010BeamlineProposalResults.asp>

Keywords: macromolecular_synchrotron_X-ray_crystallography, synchrotron_radiation_source, synchrotron_X-ray_instrumentation

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Construction of new time-of-flight single crystal diffractometer SENJU at J-PARC

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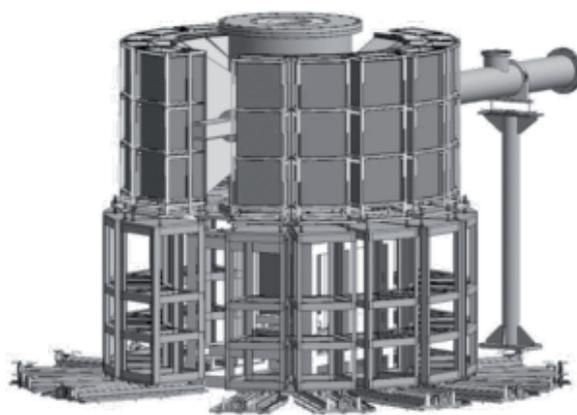
A new time-of-flight single crystal diffractometer, SENJU, is being constructed in Materials and Life Science Experimental Facility (MLF) at Japan Proton Accelerator Research Complex (J-PARC). This instrument aims to pursue precise crystal and magnetic structure analyses including local structures. Target materials will be inorganic and organic materials with lattice constants up to 50 \AA . The measurable sample size will be 0.1 mm^3 on account of the high flux neutrons from the pulsed source. A wide spectrum of the pulsed neutron, together with wide coverage of scattering angles up to 4 sr, also makes possible the observation of Bragg reflections in a wide reciprocal space at once, which eases detections of clues of phase transitions such as super

lattice reflections. In addition, a nearly symmetrical peak shape owing to a poisoned decoupled moderator will realize accurate analyses of diffuse scattering. The diffracted neutrons will be detected with newly developed scintillation detectors (256 x 256 mm^2) with the spatial resolution of 4 mm.

Available sample environments will include low temperature, high magnetic field and high pressure. A superconducting magnet has a wide opening angle for diffracted neutrons and a large bore around sample area. A dilution refrigerator (~ 50 mK) can be mounted onto the magnet. Other ancillary equipment can be also utilized in combination with another.

Softwares were also developed based on the software "STARGazer" that has been developed for iBIX at J-PARC. New features such as controlling goniometers and ancillary equipment, live-monitoring of measurements and the reconstruction of a intensity distribution in 3D reciprocal space were introduced.

Although the schedule has been delayed because of the devastating disaster, SENJU will be in commission in 2012.



Keywords: single crystal, structure, neutron,

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New developments for the phase 1 macromolecular crystallography beamlines at diamond light source

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Diamond Light Source [1] is the UK third generation synchrotron facility located south of Oxford. In the first Phase the structural biology community was served by the macromolecular crystallography (MX) beamlines I02, I03 and I04 starting with the user programme in early 2007. These widely tuneable (5-25 keV) SAD/MAD beamlines were complemented in Phase 2 with a MAD capable microfocus beamline I24 (7-25 keV) and a fixed-wavelength high-throughput station I04-1 (13.53 keV). In Phase 3 the long wavelength beamline I23 (3-12 keV), which is in the planning and construction stage, will complement the MX beamline portfolio [2].

High quality results, with over 520 structures submitted to the PDB, have been obtained from the Phase 1 MX beamlines during their operation so far. In order to improve efficiency we have improved the automation system, including a quicker sample exchange with the sample transfer robot and automatic loop finding and centering procedures. All three beamlines can now also be fully operated

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remotely. Beamline I03 has recently been upgraded with a Pilatus 6M detector running at 25 Hz. This leads to an increase in throughput and allows for new methods like faster grid scans for locating hardly visible samples or to find the best area of a larger sample. We also provide data collection strategies and crystal and diffraction image characterization automatically. Very shortly after the data collection has finished the results from our automatic data processing pipeline are available and we have extended this now to the generation of difference electron density maps if a suitable PDB file is provided.

In order to adapt to the future scientific requirements of the structural biology community we are in the process of installing new experimental end-stations on the Phase 1 beamlines. The first of these has recently been installed on beamline I04 and details will be presented elsewhere.

In addition to the beam delivery by a bimorph KB mirror system providing typical beam sizes of 90 μm x 30 μm over the complete energy range, the new end-station is also equipped with two sets of compound refractive lenses (CRL) providing a beam size of 10 x 4 microns. This presents an additional challenge on the performance of the collimation system components, especially beam diagnostics for beam intensity and position measurements. Some new developments and preliminary results will be discussed. The new end-station also provides the possibility to add a mini kappa goniometer head and preparation work is ongoing for this.

Future capabilities will include category 3 pathogenic sample handling (I03) and an adaptable and improved software user interface. An update on these developments will also be presented.

[1] <http://www.diamond.ac.uk> [2] <http://www.diamond.ac.uk/Home/Beamlines/MX.html>

Keywords: diamond light source, macromolecular crystallography, beamlines

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Structural evolution of poly(ether-*b*-amide) elastomers during uniaxial drawing studied using in-situ synchrotron WAXS and SAXS

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Poly(ether-*b*-amide)s have been commercially known as PEBAX. The general structural formula of these block copolymers is HO-[CO-PA-COO-PE-O]-H where PA and PE are polyamide and polyether blocks, respectively. Atochem[®] uses nylon 12 and poly(tetramethylene oxide) (PTMO) for PA and PE blocks, respectively, for a PEBAX series with trade names of PXX33 where XX represents the amount of the PA measured by hardness of the block copolymer.[1-2] Structural evolution of PEBAX elastomers during uniaxial drawing was studied using in-situ WAXS and SAXS for elucidating hierarchical morphological development using a synchrotron radiation source with two distinctive block copolymers having different amounts of the soft and hard segments, P6333 and P2533 which represents a soft elastomer and a hard rubber, respectively. The in-situ SAXS and WAXS tracked morphological change of the lamellar, the crystal structure of nylon 12 block, and strain-induced crystallization of the polyether block. High flux of x-rays at a synchrotron made it possible to acquire structural information during sample stretching in real time which was beneficial over the methods used in the past by holding the samples at specific strain.

Several kinds of the nylon 12 crystal such as γ , α , α' , and α'' were observed at the different draw ratios during drawing PEBX film. P2533 has much longer polyether block than P6333. Long polyether block of P2533 could cause not only the strain-induced crystallization but also the fibrillation of the stretched chains. Short polyether block of P6333 prohibited the strain-induced crystallization and transferred the stretching force into the lamellae of the nylon 12 crystal so that the anisotropic crystal lattice deformation was observed only for P6333.

[1] G. Deleens, P. Foy, E. Marechal, *Eur Polym J* **1977**, *13*, 337. [2] G. Deleens, ANTEC **1981**, 419.

Keywords: PEBAX, synchrotron, elastomer

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Facilities for Macromolecular Crystallography at BESSY II – HZB Berlin.

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The Macromolecular Crystallography (MX) group at the Helmholtz-Zentrum Berlin (HZB) has been in operation since 2003. Since then, three state-of-the-art synchrotron beam lines (BL14.1-3) for MX have been built up on a 7T-wavelength shifter source [1,2]. Currently, the three beam lines represent the most productive MX-stations in Germany, with more than 500 PDB depositions. BLs14.1 and 14.2 are energy tunable in the range 5.5-15.5 keV, while BL14.3 is a fixed-energy side station (13.8 keV). All three beam lines are equipped with CCD-detectors. Beam lines BL14.1 and BL14.2 are in regular user operation providing about 200 beam days per year and about 600 user shifts to approximately 50 research groups across Europe. BL14.3 has been equipped with a HC1 crystal dehydration device and has been set back to user operation as a screening and test beam line in 2010. BL14.1 has recently been upgraded with an MD2-microdiffractometer including a kappa-geometry option and an automated sample changer. Additional user facilities include office space adjacent to the beam lines, a sample preparation laboratory, a biology laboratory (safety level 1) and high-end computing resources. On the poster, a summary on the experimental possibilities of the beam lines and the provided ancillary equipment for the user community will be given.

[1] U. Heinemann, K. Büsow, U. Mueller, P. Umbach, *Acc. Chem. Res.* **2003**, *36*, 157-163. [2] U. Mueller *et al.*, in preparation.

Keywords: synchrotron, beam line, macromolecular crystallography

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Colliding Beam Anomalous Measurements for S and P Phasing

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