## MS07.P09

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**Ultra-fast dynamics of Silver behenate investigated by FEL femtosecond radiation (FLASH)**

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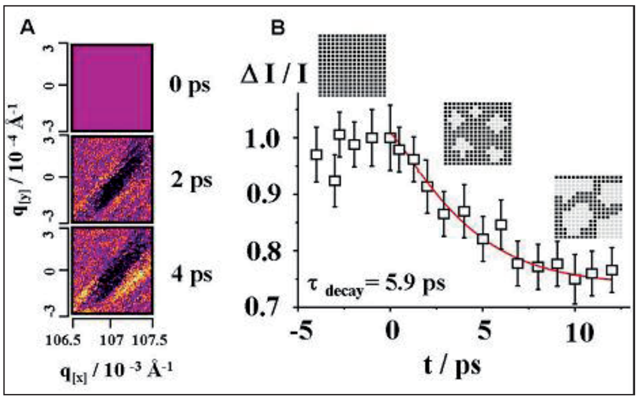
It has been proposed that free electron laser radiation can be used for ultrafast time-resolved x-ray diffraction experiments based on the NIR pulse / FEL probe scheme. We developed a multipurpose vacuum chamber which function is to be used in the pump / probe diffraction experiments with free electron laser (FEL) radiation.

By exciting with optical laser pulses in the near infrared regime (800 nm) it is possible to investigate in silver behenate (AgBh) an optical excitation dependent intensity modulation of its Bragg reflection. The time-resolution of the experiment was about 150 fs. It is assumed that in AgBh the optical laser pulses introduce a structural strain and disorder phase which can be monitored by the intensity modulation of the AgBh Bragg reflection. The strain / disorder results from an ultrafast temperature jump in the system through the non-resonant excitation at 800 nm.

Our studies on model systems like silver behenate has answered the questions of whether it is possible to use soft x-ray free electron laser radiation for studying chemical systems of periodic order and whether it is possible to utilize the time structure of soft x-ray free electron laser radiation for studying the structural dynamics of chemical systems of periodic order, opening a window for the structural studies on nano-periodic systems [1], [2].




Time dependent changes of the Bragg diffraction peak intensities (photo-excited AgBh nanotubes)

A) Intensity difference maps for various time points

B) Time evolution of the integral intensity changes of the Bragg reflection. The creation of the photo-disordered phase and its modelbased on its kinetic analysis are shown in the inset

[1] I. Rajkovic, W. Quevedo, S. Techert, et. al, *Physical Review Letters* **2010**, *104*, 125503. [2] H. Chapman, A.P. Holl, et. al, *Nature,* **2011,** *470*, 73-77.

**Keywords: free electron laser, silver behenate, and structural dynamics**

## MS07.P10

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**iMATERIA, the versatile neutron diffractometer at J-PARC -the current status of iMATERIA -**

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Ibaraki prefecture, the local government of the area where J-PARC sites in Japan, has decided to build a versatile neutron diffractometer (IBARAKI Materials Design Diffractometer, iMATERIA [1]) to promote industrial applications for neutron beam in J-PARC. iMATERIA is planned to be a high throughput diffractometer so that materials engineers and scientists can use this diffractometer like the chemical analytical instruments in their materials development process. It covers the d in range 0.18 < d (Å) < 5 with Δd/d = 0.16 % at high resolution bank, and 5 < d (Å) < 800 with the resolution changing gradually at three detector banks of 90 degree, low angle and small angle. So, this diffractometer covers very wide d-range (0.18 < d (Å) < 800). It takes several minutes to obtain a 'Rietveld-quality' data for the X-ray laboratory sized sample measured at 1MW. Currently, the beam power is limited for tuning the accelerator (~200kW), so that the measuring time is about 15 to 30 min for standard oxide samples. To promote industrial applications, a utilization system of this diffractometer is required. Since several tens to thousands experiments will be carried out in one year, we have prepared an automatically sample exchange system and large numbers of sample holders. The analysis software is also very important for powder diffraction data, so that we prepare a software package consisting of combination of several powder-diffraction software, include Rietveld analysis software (Z-Rietveld [2]), structural databases and visualization. The construction of iMATERIA was completed and user program was already started since June 2009 for high resolution bank. Because of big earthquake (the 2011 off the pacific coast of tohoku earthquake) in Japan, iMATERIA was also damaged. Currently, restoration workes is progressing. The current status and the recent data of iMATERIA include low angle bank and small angle bank will be reported.

[1] T. Ishigaki et. al., *Nucl. Instr. Meth. Phys. Res. A,* **2009,** *600,* 189-191. [2] R. Oishi, et. al., *Nucl. Instr. Meth. Phys. Res. A,* **2009,** *600,* 94–96.

**Keywords: neutron, diffractometer, powder diffraction**

## MS07.P11

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**Towards macromolecular crystallography beamlines at NSLS-II**

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Anticipating the unprecedented brightness and flux at the National Synchrotron Light Source-II, we are exploring methods and techniques that will benefit crystallographers to optimally use an initial complement of specialized instruments for macro-molecular crystallography.

At the micro-focusing beamline, FMX, with a beam size of 1–20 µm, one experimental challenge will be the routine delivery of small crystals into the focal spot. Intended to receive the most challenging samples, the beamline may also support acoustic ejection of droplets with passenger crystals onto meshes, followed in the beam by grid search, which has been shown by several of us to be a highly effective micro-crystal handling method.

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 $5x10^{-5}$) 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.

This work is supported by the NCRR and NIGMS of the US National Institutes of Health, the OBER of the US Department of Energy, and the New York Structural Biology Center.

[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**

## MS07.P12

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

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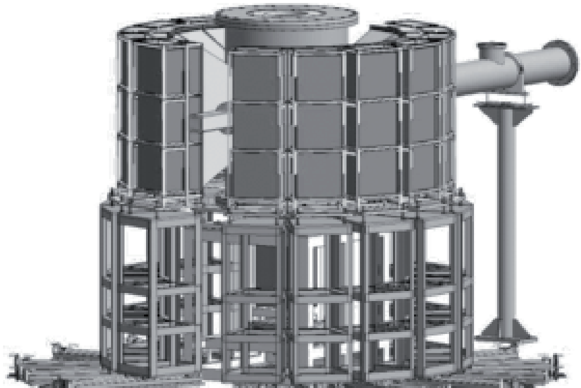
A new time-of-flight single crystal diffractometer, SENJU, is being constructed in Materials and Life Science Experimental Facility (MLF) at Japan Proton Accelerator Research Complex (J-PARC). This instrument aims to pursue precise crystal and magnetic structure analyses including local structures. Target materials will be inorganic and organic materials with lattice constants up to 50 Å. 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,**

## MS07.P13

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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