

## **1. Status of Beamline BL07LSU at SPring-8**

The University-of-Tokyo high-brilliance synchrotron soft X-ray outstation beamline BL07LSU at SPring-8 has been maintained by the permanent staff members with adjuncts for user operations. The scientific aim of the beamline is to promote advanced spectroscopy for solid state and soft (including bio-) materials. There are four regular endstations for time-resolved soft X-ray spectroscopy (TR-SX spectroscopy), ambient pressure X-ray photoelectron spectroscopy, 3D-scanning photoelectron microscope (3D nano-ESCA) and high-resolution soft X-ray emission spectroscopy (HORNET) that are open for users. There is also a free port station for users who bring their own experimental apparatus.

The beamline BL07LSU is equipped with a segmented cross-type undulator. Circularly and linearly polarized soft X-rays can be used in user experiments at full energy range (250 – 2000 eV). By using phase shifters between the neighboring undulator segments, polarization of the soft X-ray beam is regulated continuously and the switching frequency can be tuned up to 13 Hz. At the end-stations, various scientific experiments were carried out by both the laboratory staffs and users with applications of G-type (general), S-type (special), and P-type (priority). A user group of the S-type spends the long-term beamtime to pursue the challenging issues in synchrotron radiation research. A user team of the P-type proceeds an experiment by utilizing the advanced light source, especially high-speed polarization switching, of BL07LSU and by developing beamline and endstation technologies for the next generation light source.

It should be mentioned that the beginning of the year 2020, there has been a serious coronavirus (COVID-19) pandemic over the world and care has been urgently needed for those affected. In living, we have had to stop the spread of the virus by avoiding three Cs: closed spaces, crowded places, and close contact. No exception has been applied for experiments in any facilities or laboratories, including our beamline, SPring-8 BL07LSU. A number of the allocated beamtimes were split, canceled or postponed. However, the users were patient and worked their bests to proceed their research at each end-station, as briefly described below.

### **(1) Time-Resolved soft X-ray spectroscopy station (TR-SX spectroscopy)**

The station is to perform time-resolved photoemission spectroscopy experiments by synchronizing the high-brilliant soft X-ray and the ultra-short laser pulses. A two-dimensional angle-resolved time-of-flight (ARTOF) analyzer is equipped for the efficient time-resolved measurements and the sample temperature can be controlled from 15 K to 1150K. The station adopts two different pumping laser systems synchronized with the

soft X-ray beam; low repetition rate and high pulse energy (1 kHz, mJ) and high repetition rate and low pulse energy (208 kHz,  $\mu$ J).

In 2020, the experimental chamber and the ARTOF analyzer was transferred to a beamline of the vacuum ultraviolet/soft X-ray lasers (high-harmonic generation lasers) at Kashiwa campus in the University of Tokyo. This is because to improve temporal resolution of the system from picoseconds to femtoseconds for tracking the ultrafast dynamics that have not been observed at synchrotron radiation facilities yet. The last users were members of the Kagomiya group and they have succeeded in tracing dynamics of oxygen atoms during the current flow through the oxygen permeable membrane (*operando* experiment).

## **(2) Ambient-pressure X-ray photoelectron spectroscopy (AP-XPS)**

AP-XPS station allows *operando* observation of catalysts under reaction conditions. The AP-XPS station was constructed in 2014 by an external funding (JST ACT-C project), and it became opened to external users since 2018. The AP-XPS system is equipped with a differentially pumped electron analyzer (SPECS, PHOIBOS 150 NAP) and an ambient-pressure gas cell. XPS measurements can be performed both under ultrahigh vacuum and in near-ambient gas pressure up to 20 mbar. Catalytic activity is monitored by mass spectrometer, and simultaneous evaluation of adsorbate and catalyst electronic states is performed. In addition to the experiments using synchrotron soft X-ray, off-line experiments can be performed by using a twin-anode X-ray source.

In 2020, a variety of research projects have been conducted at the AP-XPS station, for example, redox reaction on nanoparticles of transition metal, coupling reaction on model catalysts of metal/metal-oxides, and development of time-resolved AP-XPS.

## **(3) 3D-scanning photoelectron microscope (3D-nanoESCA)**

3D-nanoESCA can be used for sub-100 nm range microscopic 2D mapping and depth profile of the chemical structure of functional materials and devices. In 2020, in collaboration with Drs. Asakura and Hosono at AIST group, chemical state mapping for Li and Ti was conducted on the  $\text{Li}_4\text{Ti}_5\text{O}_{12}$  cathode of Li-ion battery (LIB) to elucidate two Li phases by local Li insertion/extraction reactions. Operando observation of Li domain wall migration in LIB was also carried out. Observation of solid electrolytes in all-solid-state batteries is also a challenge, and cooling trap mechanism is being developed to prevent adsorption of volatiles from X-ray irradiated samples.

## **(4) Ultra high-resolution soft X-ray emission spectroscopy (HORNET)**

The station is dedicated for soft X-ray emission (or resonant inelastic X-ray scattering: RIXS) spectroscopy measurements with high-resolution ( $E/\Delta E > 10,000$ ) and under various environmental conditions (gas, liquid, and solid).

In 2020, 13 proposals were accepted, and 4 proposals (Resonant inelastic soft X-ray diffraction of nanoparticles, elucidation of mechanism of induced structural change in thermo-responsive polymers, and observation of water alignment in polyelectrolyte membranes) were carried out by internal staff. The other nine projects were mostly carried out in the latter half of 2020 due to cancellation of user operation by the coronavirus pandemic. As one of the anti-coronavirus activities, RIXS was used to evaluate the function of biocompatibility on the surface of the inner tube of extracorporeal membrane artificial lung (ECMO) coated by poly(2-methoxyethyl acrylate) (PMEA) to inhibit thrombus formation. The result was used to synthesize more excellent biocompatible polymers, which was introduced in Nikkei newspaper and the paper has been submitted to Nature Communications.

Feasibility studies of five major companies were carried out using part of the beamline adjustment time for the next-generation synchrotron radiation.

#### **(5) Soft X-ray imaging (Free-port station)**

We have started to develop a microscopic imaging system using Wolter mirrors in the latter half of 2020 for use in the next-generation synchrotron radiation facilities. In collaboration with a company, we have already developed and evaluated a 200-mm-long large Wolter mirror. By introducing a mirror with a much larger aperture than conventional ones, we aim to develop a system that enables imaging with a minimum resolution of 50 nm. The main targets of the system are scanning X-ray microscopy and typography using a focusing profile without small chromatic aberrations. We also plan to use the long working distance of the soft X-ray mirror to perform tomography and operando measurements.