# 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. A scientific aim of the beamline is to promote advanced spectroscopy for solid state and soft (including bio) materials. There are currently four regular endstations: time-resolved soft X-ray spectroscopy (TR-SX spectroscopy), soft X-ray diffraction, 3D-scanning photoelectron microscope (3D nano-ESCA) and ultrahigh 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. By using phase shifter among the undulator segments, a polarization control of soft X-ray was started since 2016. Circularly and linearly polarized soft X-rays at full energy range (250 – 2000 eV) have been available by tuning the permanent magnet type phase shifter. Additionally, fast circular polarization switching at a rate of 10 Hz and 13 Hz has been achieved at the photon energy, requested by users. In 2017, a new technique of the magneto-optical experiment was developed by using this polarization control.

At the beamline endstations, various scientific researches were carried out by both the laboratory staffs and general users (G-type application). Below are brief introduction of recent activities at each station.

# (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 twodimensional angle-resolved time-of-flight (ARTOF) analyzer has been equipped for the efficient time-resolved measurements and the measurement temperature can be controlled from 15 K to 1150K. The station adopts high repetition rate (208 kHz) laser that successfully synchronizes with synchrotron radiation soft X-rays.

In 2017, measurements of two-dimensional photoelectron diffraction patterns were performed by taking advantage of the ARTOF detector. Patterns from the individual chemical shifts show the difference and, thus, contain structural information at their atomic sites. The time-resolved measurement is expected to trace atomic motion in the dynamical events.

Photocatalytic activity and lifetime of photoexcited carriers on well-defined singlecrystalline anatase and rutile TiO2 surfaces with different surface orientation have been systematically studied by static and dynamic photoelectron spectroscopy. Photocatalytic activity, evaluated with reference to the photocatalytic degradation of acetic acid, has a positive and linear correlation with carrier lifetime at the crystal surface, which was determined by following the time evolution of the ultraviolet-induced surface photovoltage. The carrier lifetime was found to be a prime factor for the photocatalytic activity.

### (2) 3D-scanning photoelectron microscope (3D nano-ESCA)

An originally developed system "3D-nano-ESCA", which is a combination of soft X-ray scanning photoelectron emission microscopy (SPEM) and an angle-resolved electron analyzer, is installed in this station. Users can use 3D-nano-ESCA for sub-100 nm range microscopic 2D mapping and depth profile of the chemical structure of functional materials and devices.

In 2016, operando nano-spectroscopy was performed for GaN-HEMT devices as collaboration with Sumitomo Electric Industries, Ltd. and Tohoku University as part of the NEDO academic-industrial alliance project. Almost 10 V shift of the Ga 3d level observed by application of a voltage between the gate and the drain of the transistor was successfully explained by the presence of interface state with a doping level of 10<sup>13</sup> cm<sup>-2</sup> at the AlGaN/GaN interface, which was found to be the origin of the current collapse effects in GaN-HEMT. Moreover, reduction of the interface state was observed as a reduction of the Ga 3d peak shift by an epitaxial growth of a 3 nm SiN capping layer on the GaN surface. Another operando experiments were performed on a cathode material LiNi<sub>0.5</sub>Mn<sub>1.5</sub>O<sub>4</sub> of a Li-ion battery as collaboration with AIST. Changes in the local electronic structure of LiNi<sub>1.5</sub>Mn<sub>0.5</sub>O<sub>4</sub> after charging/discharging were analyzed to elucidate detailed charge redistribution upon Li-ion exertion/insertion.

#### (3) 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 ultra high-resolution ( $E/\Delta E > 10^4$ ) and under various environmental conditions (gas, liquid and solid). The number of applications to the HORNET station is increasing. In 2017 we have performed 17 collaborative works using the HORNET station, four of which are related to the study on the behavior of water at various circumstances (water in plasma, water encapsulated in an electrolyte, hydrated water, and interfacial water). We also published four papers related to water. RIXS in operando conditions were frequently used for the study of fuel cell batteries and rechargeable Li- and Na-ion batteries. Angle (momentum) resolved system was also utilized for the oxygen analyses of strongly correlated systems like multilayered high T<sub>c</sub>

cuprate Bi2223 and other hole-doped cuprates, as well as for the soft X-ray inelastic diffraction (SXID) of LaSrFeO<sub>4</sub> by collecting the data more precisely than preliminary results in the previous year. For future studies on water related materials and SXID verification a liquid jet system was installed which removed the vacuum compatible membrane separating atmospheric pressure and high vacuum expecting more precise and quantitative analysis of the intensity of elastic scattering, which may also develop a new field that combines spectroscopy and diffraction in the soft X-ray region. Continuous studies on bio-inspired or bio-model compounds are ongoing, patiently and systematically controlling radiation damage problems and some of them (model compounds of Fe-S proteins, Mn cluster in photosystem II) have already been on the publication stage.

## (4) Free-port station

The station is equipped with a focusing mirror chamber, and users can connect their own experimental chambers. In 2017, the following experiments were performed: time resolved soft X-ray diffraction of Ba<sub>3</sub>CuSb<sub>2</sub>O<sub>9</sub>; time resolved XAS of Eu compounds with valence fluctuations; ambient-pressure X-ray photoemission spectroscopy of  $H_2$  adsorption/absorption in Pd alloys and CO<sub>2</sub> hydrogenation reactions on Ni-based catalysts; improvement of high-energy-resolution two-dimensional photoelectron spectrometer DELMA (display-type ellipsoidal mesh analyzer) to measure clear 2-pi hologram at low-temperature.

# References of the individual researches are listed as publications in different pages.