## BL25SU Soft X-ray Spectroscopy of Solid

#### 1. Introduction

BL25SU is dedicated to soft X-ray spectroscopic studies on the electronic and magnetic states and the surface structures of solids. After a major upgrade in FY2014, the beamline now has two branch lines. The A-branch supports high-energy resolution measurements, while the B-branch is optimized for nano-focused beams with a small-angle divergence <sup>[1-3]</sup>. The present status of the main beamline components is described below.

# 2. Status of the beamline and experimental apparatuses

#### 2-1. Beamline

Because soft X-ray absorption spectroscopy provides opportunities to measure the K-absorption edges of oxygen and nitrogen, a high-flux soft Xray beam with a low energy is necessary. In FY2019, a new varied-line-spacing plane grating with a grooving density of 300 line/mm was installed in the B-branch. In addition, the existing gratings and focusing mirrors were ozone-cleaned as it is well known that they are severely contaminated in the early stage after the vacuum chamber activation. The new grating covers not just the *K*-absorption edges of nitrogen and oxygen but also the Labsorption edges of all 3d transition elements. The high flux is expected to shorten the measurement time and reduce the degradation of data accuracy with time.

# 2-2. Two-dimensional photoelectron spectroscopy (A-branch first station)

The apparatus has an analyzer, which can measure

the wide-angle distribution of photoelectrons. Using this apparatus, unique methods such as surfacesensitive photoelectron holography <sup>[4]</sup>, atomic orbital analysis by circularly polarized resonance photoelectron diffraction <sup>[5]</sup>, and microscopic photoelectron diffraction <sup>[6]</sup> have been developed. Due to continuous maintenance, the apparatus is stably operating.

## **2-3.** Retarding field analyzer (RFA) (A-branch second station)

Photoelectron diffraction (PED) or photoelectron holography allows non-periodic local structures with multiple chemical states to be studied <sup>[7]</sup>. These methods require wide-range photoelectron angular distribution patterns measured with a sufficiently high energy resolution to resolve core level chemical shifts. For such measurements, a displaytype retarding field analyzer (RFA) with a high resolving power ( $E/\Delta E$ ) of 1100 was developed <sup>[8]</sup>. The retarding grid is a wire mesh, but simulations predicted that the energy resolution can be further improved by a retarding grid composed of cylindrical holes <sup>[8]</sup>.

To maximize the energy resolution, the magnetic field in the RFA must be minimized. The previous vacuum chamber on which the RFA was mounted was composed of stainless steel and lacked a magnetic shield function. At the end of FY2019, the chamber was replaced with a  $\mu$ -metal chamber, which serves as a magnetic shield. The energy resolution of an RFA with a cylindrical hole grid will be examined in FY2020.



Fig. 1. RFA station constructed at the A-branch of BL25SU.

**2-4. Microbeam angle-resolved photoemission spectroscopy (ARPES) (A-branch third station)** The ability of selecting flatly cleaved areas from poorly cleaved sample surfaces is valuable for ARPES <sup>[9]</sup>. To enhance this capability, a micro-ARPES end-station equipped with a DA30 analyzer from Scienta Omicron and a micro focusing mirror was developed <sup>[10]</sup>. The smallest available beam spot is 5 μm. This end-station was opened for public use in FY2018.

In FY2019, one user group planned experiments involving small crystals, which were about 10  $\mu$ m in size. Because the probing depth is a few nanometers, ARPES requires clean sample surfaces. Typically, samples are cleaved *in situ* to obtain clean surfaces. However, cleaving 10- $\mu$ m crystals is challenging. To obtain clean surfaces, an *in situ* sample-heating system was set up in the preparation chamber of the micro-ARPES end-station. The temperature can reach more than 1000 °C. In FY2019, this system was successfully used for the user experiment.



Fig. 2. Micro-ARPES station constructed at the Abranch of BL25SU.

### 2-5. Pulse magnet-type X-ray magnetic circular dichroism (XMCD) spectroscopy (B-branch first station)

In an experiment using the pulse magnet-type XMCD apparatus, a long interval time of about 20 min was required to generate 40 T and to cool the pulse magnet after each pulse generation. An X-ray shutter linked to the measurement system was installed to avoid unnecessary X-ray irradiation, which induces sample damage. In addition, this station has a mechanism to retract this apparatus. Depending on the purpose, various types of equipment can be temporarily installed.

## **2-6.** Electromagnet-type XMCD spectroscopy (B-branch second station)

This apparatus can selectively use low-temperature,

high-temperature, and voltage/current application measurements. Combining these methods with total electron yield (TEY), partial fluorescence yield (PFY), and transmission modes allows for diverse experimental environments. In FY2019, a heating system was installed in the sample preparation chamber to enable sample annealing up to 400 °C.

#### 2-7. Scanning soft X-ray microscope (nano-XMCD) (B-branch third station)

A scanning soft X-ray microscope was developed with the support of the Elements Strategy Initiative Center for Magnetic Materials (ESICMM) funded by the Ministry of Education, Culture, Sports, Science and Technology (MEXT) of Japan<sup>[11]</sup>. This unique apparatus features nanoscale MCD imaging under high magnetic fields. It is intensively used to study permanent magnets, where magnetic fielddependent changes in the magnetic domains clearly microscopic visualize the origin of the process<sup>[12]</sup>. In demagnetization general, а coercivity of magnetic materials decreases under high temperature. To investigate this mechanism in detail, a sample-heating system was developed (Fig. 3) in FY2019. In FY2020, magnetic imaging of permanent magnets will be tried at a sample temperature of 200 °C or higher.



Fig. 3. New high-temperature sample stage for nano-XMCD at the B-branch of BL25SU.

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#### **References:**

- [1] Nakamura, T. et al. (2014). SPring-8 INFORMATION, 19, 102-105.
- [2] Nakamura, T. et al. (2015). SPring-8/SACLA Research Report, 3(1), 186-200.
- [3] Senba, Y. et al. (2016). AIP Conference Proceedings, 1741, 030044.
- [4] Matsui, F. et al. (2016). Sci. Rep. 6, 36258.
- [5] Matsui, F. et al. (2015). *Phys. Rev. Lett.*, 114, 011501.
- [6] Sugita, K. et al. (2016). *e-J. Surf. Sci. Nanotechnol.*, 14, 59.
- [7] Tsutsui, K. et al. (2017). Nano Lett. 17, 7533.
- [8] Muro, T. et al (2017). *Rev. Sci. Instrum*, 88, 123106.
- [9] Fujiwara, H. et al. (2015). J. Synchrotron Rad. 22, 776.
- [10] Senba Y. et al. (2020). J. Synchrotron Rad., 27, 1103 (2020).
- [11] Kotani, Y. et al. (2018). J. Synchrotron Rad., 25, 1444-1449.
- [12] Billington, D. et al. (2018). *Phys. Rev. Materials*, 2, 104413.