BL17SU
RIKEN Coherent Soft X-ray Spectroscopy

1. Introduction

Recently, we have been operating the soft X-ray (SX) undulator beamline BL17SU mainly for SX microspectroscopy. BL17SU was originally constructed in 2004 to advance spectroscopic studies for materials science by means of conventional X-ray absorption spectroscopy (XAS), X-ray photoemission spectroscopy (XPS) and X-ray emission spectroscopy (XES). In the early phase of BL17SU, we also started the operation of the spectroscopic photoemission low-energy electron microscope (SPELEEM, ELMITEC GmbH) [1] for public use in addition to the operation for in-house staff. Since then, it has been providing opportunities to study the local electronic structure as well as the magnetic domain structure of advanced materials with a spatial resolution of about 22 nm.

2. Recent activities

To extend further the research opportunity of BL17SU to the microspectroscopic study of various kinds of materials, we installed a versatile photoemission electron microscope (PEEM, FOCUS GmbH) into the carry-in station of the BL17SU b-branch at the end of FY2016. After the commissioning of the versatile PEEM, we achieved a spatial resolution of about 37 nm for the versatile PEEM when a UV lamp was used (approximately 100 nm when using SR) as the excitation source. At the beginning of FY2018, we opened the apparatus to public users. We have also developed a time-resolved PEEM measurement system using the versatile PEEM combined with a femtosecond laser system and an SX chopper [2]. Advanced research using this system to investigate transient changes of the electronic and magnetic structures is now in progress.

We have also been carrying out the commissioning of the scanning SX spectromicroscope with a modest spatial resolution (*e.g.,* 300–500 nm) since the beginning of FY2018 [3]. We aim to study the local electronic structures on the surfaces and interfaces of various advanced materials under conditions ranging from low vacuum to helium atmosphere by taking advantage of the photon-in photon-out measurement scheme. Figure 1 shows a schematic illustration (a) and photographs of the apparatus (b–d) installed at the b-branch carry-in station, *i.e.*, the end-station is shared with the versatile PEEM. In Figs. 1(e)–(h), the typical results of the microspectroscopic data obtained during the commissioning phase are also shown. In this carry-in station, the two machines can be easily switched, since the machines are installed on slide rails. The scanning SX spectromicroscope, however, was moved to the a-branch of BL17SU in FY2020.

After the commissioning, we have been applying the scanning SX spectromicroscope to microspectroscopic studies of various materials by means of X-ray fluorescence (XRF) analysis. In Fig. 2, we show the results of applying microprobe SX fluorescence and absorption spectroscopic analyses to characterize the buried multilayered microstructure of permalloy fabricated on a silicon substrate [4]. We could successfully visualize μXRF elemental distribution maps as well as microprobe XAS spectra of O and Fe contained in a buried

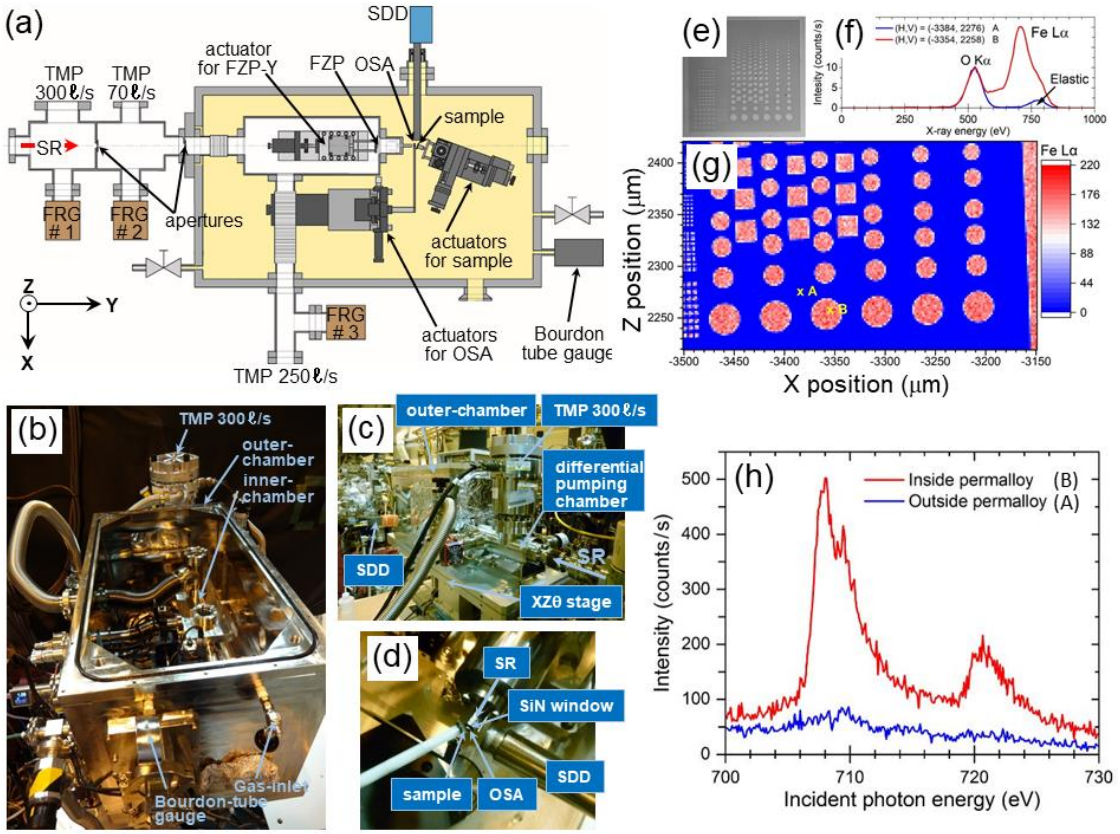

Fig. 1. (a) Top-view schematic of scanning SX spectromicroscope, where TMP is the turbomolecular pump, FZP the Fresnel zone plate, OSA the order-sorting aperture, and SDD the silicon drift detector. (b)–(d) Photographs of the present apparatus: (b) the view from downstream, (c) the view from upstream, and (d) the area around the sample. (e) Photograph of the permalloy microdot patterns fabricated on the Si substrate taken using a metallurgical microscope. (f) SX-induced XES spectra recorded inside a microdot (red curve, point B) and outside a microdot (blue curve, point A). Points A and B are marked in (g). (g) Element-specific two-dimensional mapping of permalloy microdots measured by counting the number of Fe Lα X-rays. (h) Microprobe XAS spectra recorded inside a microdot (red curve, point B) and outside a microdot (blue curve, point A) by sweeping the SX-beam energy across the Fe $L_{2,3}$-edges.

500 μm square test pattern beneath a 30-nm-thick Au coating, as shown in Fig. 2.

In Fig. 3, we show the (a) Si K-XAS and (b) XES spectra obtained from the local areas A and B marked in the single-element map (c) of an *Arachnoidiscus* sp. frustule excited using a 1860 eV focused SX-beam [5]. The single-element map was recorded by the partial fluorescence yield (PFY) method with a pixel size of 0.5 μm and an exposure time of 1 s for one pixel.

Since the end of FY2018, we have also been developing another scanning spectromicroscope whose spatial resolution is designed to be better than 30 nm at 600 eV. We began the commissioning

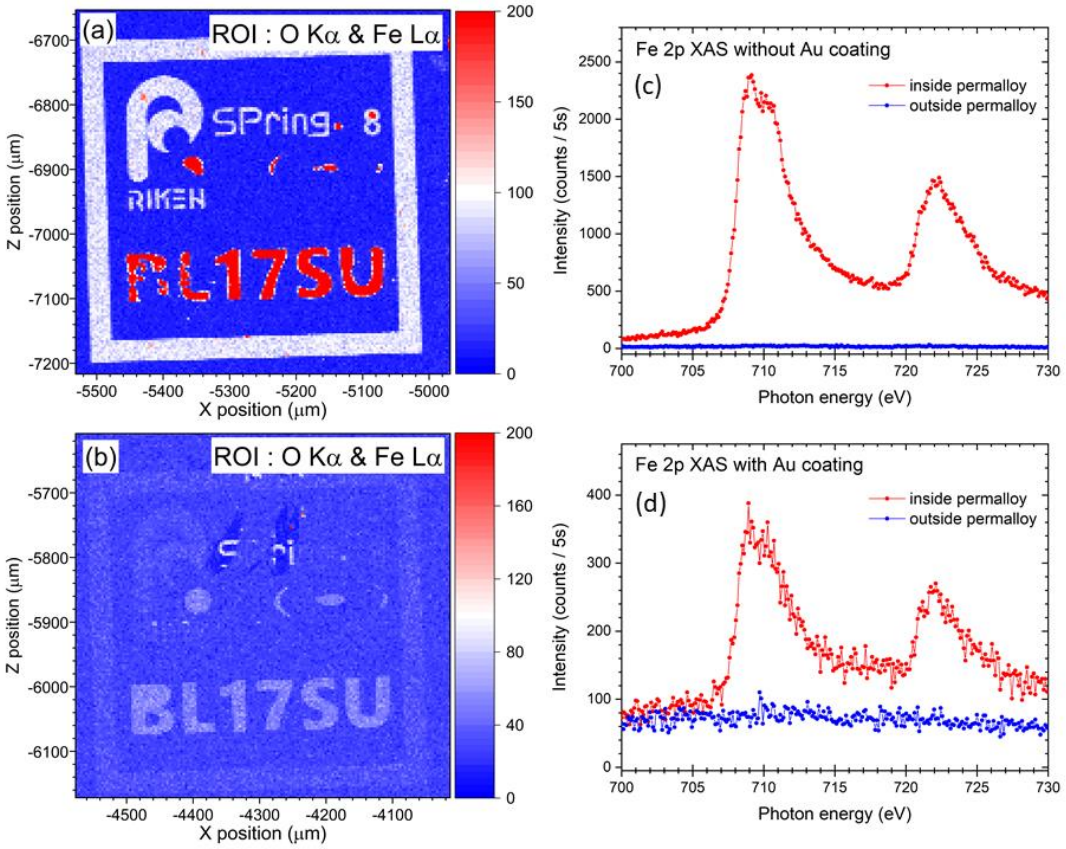

Fig. 2. (a),(b) Microprobe XRF elemental distribution maps of O and Fe in a 500 μm square test pattern visualized by measuring O Kα and Fe Lα X-rays using a 750 eV focused SX-beam without (a) and with (b) 30-nm-thick Au coating on the pattern. (c),(d) μXAS spectra recorded inside (red curve) and outside (blue curve) the "U" of the "BL17SU" pattern made of permalloy by sweeping the SX-beam energy across the Fe 2p edges with a dwell time of five seconds for one point. Measurements were carried out on the patterns without (c) and with (c) Au coating.

of the high-resolution scanning SX spectromicroscope at the beginning of FY2020. Now more than 70% of the total user time of BL17SU is devoted to microspectroscopic experiments.

Masaki Oura

Soft X-Ray Spectroscopy Instrumentation Team, Physical and Chemical Research Infrastructure Group, Advanced Photon Technology Division, RIKEN SPring-8 Center

References:

[1] Guo, F. Z., Muro, T., Matsushita, T., Wakiya, T., Ohashi, H., Senba, Y., Kinoshita, T., Kobayashi, K., Saitoh, Y., Koshikawa, T., Yasue, T., Oura, M., Takeuchi, T. & Shin, S. (2007). *Rev. Sci. Instrum.* **78**, 066107.

[2] Ohkochi, T., Osawa, H., Yamaguchi, A., Fujiwara, H. & Oura, M. (2019). *Jpn. J. Appl. Phys.* **58**, 118001.

[3] Oura, M., Ishihara, T., Osawa, H., Yamane, H., Hatsui, T. & Ishikawa, T. (2020). *J. Synchrotron Rad.* **27**, 664–674.

[4] Oura, M., Ishihara, T. & Yamaguchi, A. (2020). *Jpn. J. Appl. Phys.* **59**, 060902.

[5] Ishihara, T., Ohkochi, T., Yamaguchi, A., Kotani, Y. & Oura, M. (2020). *PLOS ONE* **15**, e0243874.

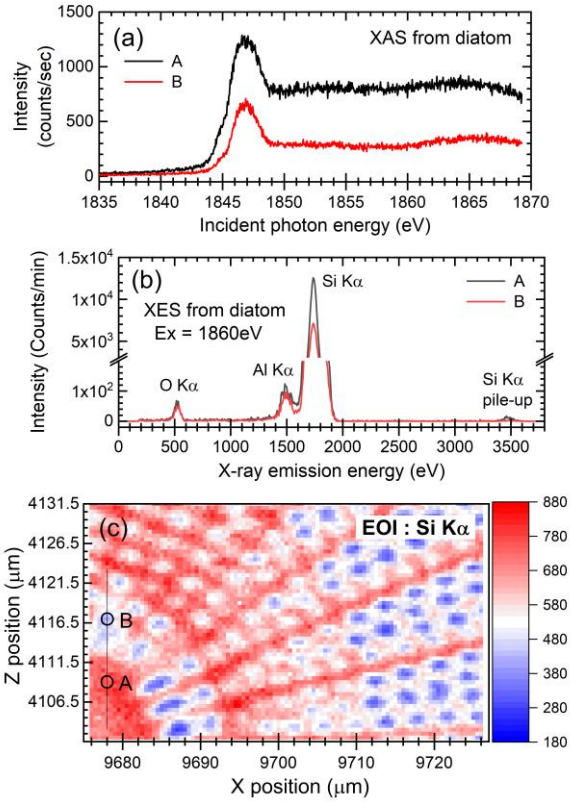

Fig. 3. (a) Si K-XAS and (b) XES spectra of a frustule of *Arachnoidiscus* sp. recorded at two different positions, A and B, marked in the (c) μXRF single-element map of Si obtained from near the center of the exterior surface of the frustule using a 1860 eV focused SX-beam. The μXRF single-element map was recorded by the PFY method with a pixel size of 0.5 μm and an exposure time of 1 s for one pixel, where EOI stands for energy of interest.