BL47XU
HAXPES･µCT

1. Introduction

BL47XU, which is an X-ray undulator beamline, is dedicated to hard X-ray photoelectron spectroscopy (HAXPES) and micro-CT. To handle the high heat load of the undulator, a liquid-nitrogen (LN2) cooling system is used to cool the monochromator crystals. The available energy range is between 6 keV and 37.7 keV with a Si(111) reflection of the monochromator. To eliminate higher harmonics, a set of reflection mirrors (double-bounce in the vertical direction) can be inserted.

The beamline has two experimental hutches (EH1 and EH2), which are located just after the optics hutch. EH1 contains an experimental table for X-ray nano-CT, while EH2 contains HAXPES and micro-CT. In FY2019, the measurement apparatuses and techniques for HAXPES and X-ray nano-CT were improved. A new wide-angle objective lens was installed for HAXPES and high-efficiency illumination optics for nano-CT was developed. This report describes the details.

2. Hard X-ray photoelectron spectroscopy (HAXPES)

An advantage of HAXPES at BL47XU is angle-resolved analysis using a wide-angle objective lens with a photoelectron acceptance angle of ±32°. It can precisely measure the chemical bonding state in a three-dimensional microdomain at a buried interface in combination with the φ1-µm–focused beam by a Kirkpatric-Baez (KB) mirror [1,2]. Although many beamlines are used for HAXPES in synchrotron radiation facilities around the world, BL47XU at SPring-8 is the only one that can perform wide-angle analysis with a micrometer-scale resolution. In addition, the above features, which allow *in situ* HAXPES measurements for samples held in an atmospheric environment cell, were developed in collaboration with a Partner User. In FY2019, the fabrication and performance of a new wide-angle objective lens were evaluated as an upgrade of the HAXPES station.

The HAXPES end-station at BL47XU is equipped with a Scienta Omicron R4000 photoelectron analyzer (Fig. 1). The standard photoelectron acceptance angle of this analyzer is ±7°. However, installing a wide-angle objective lens, which was developed at SPring-8, in the front of the analyzer in FY2019 widened the photoelectron acceptance angle of the BL47XU equipment to ±32°.

In FY2018, an insulation failure accident occurred at the high-voltage electrode of the wide-angle objective lens. A liquid sample in an atmospheric environment cell leaked and adhered to the objective lens. To restore insulation, the wide-angle objective lens had to be disassembled and cleaned. This repair took more than four days. Similar accidents, which take a long time to repair, will shorten user’s experiment time and affect subsequent experiments.

Based on this experience, another wide-angle objective lens was fabricated in FY2019 so that the lens can be replaced if future contamination occurs.

For the wide-angle objective lens to work properly, its back focal point must be aligned with the front focal point of the photoelectron analyzer. The optimal position of the objective lens relative to the analyzer is determined at the position where the photoelectron detection intensity is maximized. Therefore, the objective lens was scanned against the analyzer and the intensity of the HAXPES spectrum of gold foil was recorded. Due to the adjustment to the optimal position, the estimated energy resolution of the spectrum is 251 meV, which is similar to the value obtained with the current objective lens. Thus, the current and newly fabricated objective lenses provide comparable results. By having a backup and replacing the lens in the event of an insulation failure accident, the recovery time should be short, which will contribute significantly to the stable operation of HAXPES experiments.

Fig. 1. Wide-angle objective lens mounted on the analyzer of BL47XU.

3. Upgrade of X-ray nano-CT by installing a multiscale measurement system

The multiscale-CT system was installed at BL47XU. This system realizes both a high spatial resolution and a large field of view by combining multiple tomographic systems with different fields of view and spatial resolutions [3]. Figure 2 shows a schematic diagram. The system consists of a micro-CT, a medium-resolution nano-CT, and a high-resolution nano-CT. Table 1 lists the typical parameters of these three measurement modes.

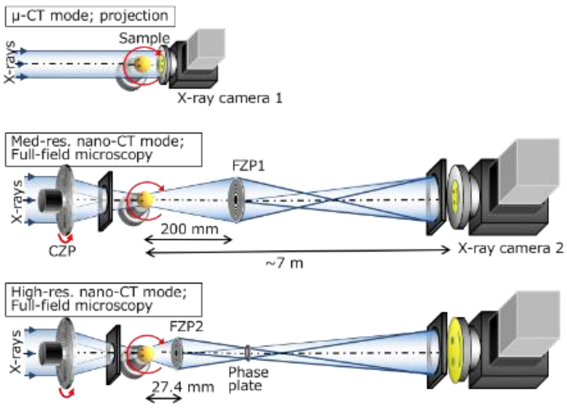

Fig. 2. Schematic diagram of multiscale-CT at BL47XU.

Table 1. Typical parameters of multiscale-CT at BL47XU

Mode	μ-CT	Nano-CT	
		Med resolution.	High resolution.
Field of view	0.8 mm	60 μm	15 μm
Resolution	~1 μm	150 nm	70 nm
Voxel size	0.5 μm	70 nm	9 nm
CT scan time (1800 projection.)	3 min	7.5 min	15 min
X-ray energy	6–37.7 keV	6–15 keV	
Contrast mode	Absorption	Absorption / Zernike phase	

Because a common sample stage is used, users can easily select one of the three measurement modes without dismounting the sample. The micro-CT using projection optics can capture the entire object. A visible-light conversion-type sCMOS camera with a typical pixel size of 0.5 μm and a field of view of approximately 1 mm is used as an X-ray camera. Two nano-CTs based on full-field X-ray microscopy using a Fresnel zone plate (FZP) objective are used for high spatial resolution measurements of the region of interest (ROI) of objects. In these two nano-CT systems, a condenser zone plate (CZP) and an X-ray camera are common.

By switching two FZPs. which have different parameters, the medium- and the high-resolution nano-CT modes are switched.

Table 2 shows the parameters of the FZPs. Although both have the same outermost zone width, the different diameters result in different focal lengths and magnification factors of the optical system.

Table 2. Parameters of FZPs.

FZP name	FZP 1 for medium resolution	FZP 2 for high resolution.
Base plate	Si 10 mm × 10 mm × 6.25 mm	
Membrane	Ru 20 nm / SiC 2 μm / SiN 0.3 μm	
Zone material	Ta	
Diameter	620 μm	85 μm
Outermost zone width	50 nm	
Thickness of inner-half zones	1 μm	
Zone number	3100	425
Focal length at 8 keV	200 mm	27.4 mm

The magnification factor, field of view, and spatial resolution of the medium-resolution nano-CT mode at 8-keV X-rays are approximately 70, 100 μm, and 200 nm, respectively. On the other hand, those of the high-resolution nano-CT mode are approximately 255, 15 μm, and 70 nm, respectively. In the nano-CT mode, the image contrast mode can also be easily selected between the absorption contrast and the Zernike phase contrast by removing or installing a phase plate at the back focal plane of the A-FZPs.

As a typical measurement example of the multiscale-CT, Fig. 3 shows CT images of a diatom fossil. The whole image was obtained with the medium-resolution nano-CT mode (Figs. 3(a) and (b)) and its ROI (surrounding region in Figs. 3(a) and (b)) was with the high-resolution nano-CT mode (Figs. 3(c) and (d)). In the region surrounded by the ellipses in Figs. 3(c) and (d), several hole-like structures with apertures of 45–60-nm diameter were resolved.

Figure 4 shows a nondestructive multiscale-CT measurement of small particles of the Orgueil meteorite, which is a type of carbonaceous chondrite. Figure 4(a) shows the CT image of the entire sample obtained with the medium-resolution mode. Figure 4(b) shows the interior CT image of the surrounding regions in Fig. 4(a) obtained in the high-resolution mode. A fibrous-like fine grained structure with a width of several hundred nanometers was clearly observed. The carbonaceous chondrite group might have originated from C-type asteroids such as RYUGU, which is the destination of the ongoing sample-return mission HAYABUSA-2 of Japan. The Orgueil meteorite was observed as an analogous case for analyzing the sample to be returned by the spacecraft mission. Because the subdivision of the sample and exposure of the ROI to the terrestrial atmosphere may cause deterioration or destruction of minerals in the sample, the effectiveness of multiscale-CT, which enables the nondestructive observations of the ROI, must be demonstrated.

By combining the high-energy X-ray types operated at BL20XU, the multiscale-CTs are routinely used in various fields, such as biology, soft materials, metallic materials, ceramics, astronomy, batteries, and devices, for nondestructive three-dimensional nano-imaging of bulky samples. Because bulky samples are much easier to be treated than tiny samples, multiscale-CT is also frequently used for four-dimensional nano-imaging such as *in situ*, *ex situ*, and operando measurements.

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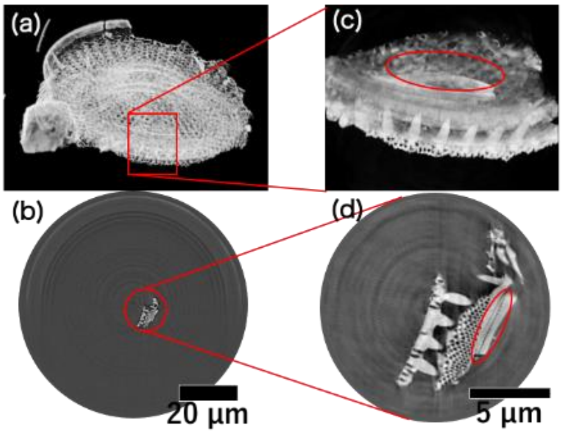

Fig. 3. Diatom fossil imaged via X-ray multiscale-CT. (a) Rendered image and (b) virtual cross-section measured in the medium-resolution nano-CT mode. (c) Rendered and (d) virtual cross-section of the surrounding region in (a) and (b) measured in the high-resolution mode.

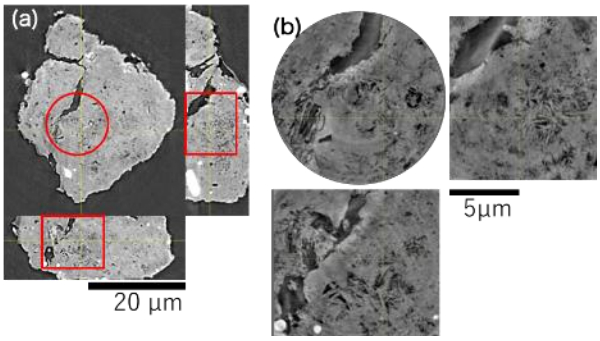

Fig. 4. Orgueil meteorite imaged via multiscale-CT from the orthogonal view. (a) Obtained in the medium-resolution mode. (b) Surrounding regions in (a) obtained in the high-resolution mode.