# BL47XU
# (HAXPES / micro-CT)

## 1. Introduction

BL47XU is an X-ray undulator beamline allocated for 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 for the monochromator crystals. The available energy range is between 6 keV and 37.7 keV with a Si(111) reflection of the monochromator. A set of reflection mirrors (double-bounce in the vertical direction) can be inserted to eliminate higher harmonics.

There are two experimental hutches (EHs) just after the optics hutch. An experimental table for X-ray nano-CT is located in EH 1. Apparatuses for HAXPES and micro-CT is located in EH 2.

As a part of the activities at this beamline, the measurement apparatus and techniques for HAXPES and X-ray nano-CT were improved. In FY2018, a high-frame-rate camera for HAXPES was installed, and high-efficiency illumination optics for nano-CT was developed. Here, the details are described.

## 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°. This enables mapping measurements of the chemical bonding state in a microdomain at a buried interface in combination with the φ1 μm focused beam by a Kirkpatric-Baez (KB) mirror [1, 2]. Many beamlines are used for HAXPES in synchrotron radiation facilities around the world, but BL47XU at SPring-8 is the only beamline that can perform wide-angle analysis with the micrometer scale resolution. In addition, the above features allow *in situ* HAXPES measurements for samples to be performed in an atmospheric environment cell (gas and liquid samples). In this section, we report updates of the charge-coupled device (CCD) detector in the photoelectron analyzer with a high-speed read time, as an upgrade of the HAXPES station in FY2018.

In the photoelectron spectrometer at BL47XU, photoelectrons converge onto the detection plane at the exit of a hemispherical analyzer with two-dimensional (2D) information of their energies and their emission positions or angles. Then, the photoelectron pulse intensities are counted by a 2D CCD detector through a fluorescent plate and a multichannel plate. Therefore, both the photoelectron analyzer and the detector performance are important for the photoelectron detection efficiency.

The 2D CCD detector previously used (Fig. 1, right) had a low reading speed of 13 fps. The accuracy of the linearity of the photoelectron detection intensity with respect to the incident light intensity was poor due to the counting loss of the photoelectron pulses, especially in high-intensity measurements. Since linearity is important to estimate elemental composition from the photoelectron intensity, users in industrial fields demanded improvements. In addition, a detector with an enhanced reading speed will improve the detection efficiency and shorten the measurement time.

Therefore, we introduced a high-speed 2D CCD detector (Fig. 1, left) with a reading speed of 70 fps

Fig. 1. Two-dimensional CCD detectors for the HAXPES analyzer. (left) high-speed detector with 70 fps, and (right) the detector with 13 fps.

in the photoelectron spectrometer. Since the number of pixels in the new detector is about half that of the old one, there were concerns about reductions in energy, position, and angular resolution. However, performance tests of angle and energy resolutions confirmed that the resolutions are comparable to those obtained with the old detector. Meanwhile, the improved detection efficiency shortens the measurement time to obtain the same S/N ratio spectrum as before. Hence, the number of data obtained within the limited machine time increased, enabling more accurate discussions. Furthermore, the enhanced detection efficiency remarkably improves the measurements for low-intensity samples such as liquid/gas experiments.

**3. Upgrade of X-ray nano-CT by installing a beam-shaping condenser zone plate**

X-ray nano-CT at BL47XU based on full-field X-ray microscope optics using a Fresnel zone plate (FZP) as an objective and a condenser zone plate (CZP) as an illuminating optics realizes 100-nm-order resolution three-dimensional imaging. This system is now widely used for various fields such as biology, medical, astronomy, mineral, material, devices, batteries, and industry. The typical scan time is approximately 20 min (0.5 s exposure, 1,800 projections). However, the scan time is too long for time-resolved measurements such as *in situ* observations. Therefore, a higher intensity of illumination is required to reduce the scan time.

A new CZP was developed to increase the illumination intensity. Figure 2a shows a critical illumination available using an FZP as a condenser. In this case, the field of view is not sufficiently large because the focused beam size is too small for a highly collimated beam from synchrotron radiation (SR) light source. Therefore, hollow-cone illumination (Fig. 2b) is commonly employed in the SR full-field X-ray microscope system. Such an illumination is realized by employing a CZP with

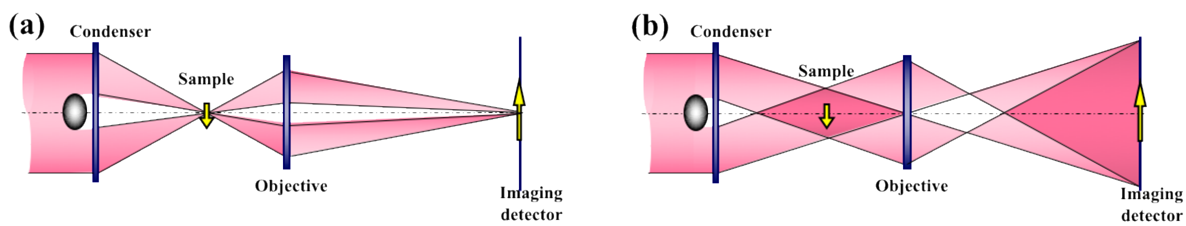


Fig. 2. Conceptual drawing of illumination for full-field microscope. (a) Critical illumination and (b) hollow-cone illumination.

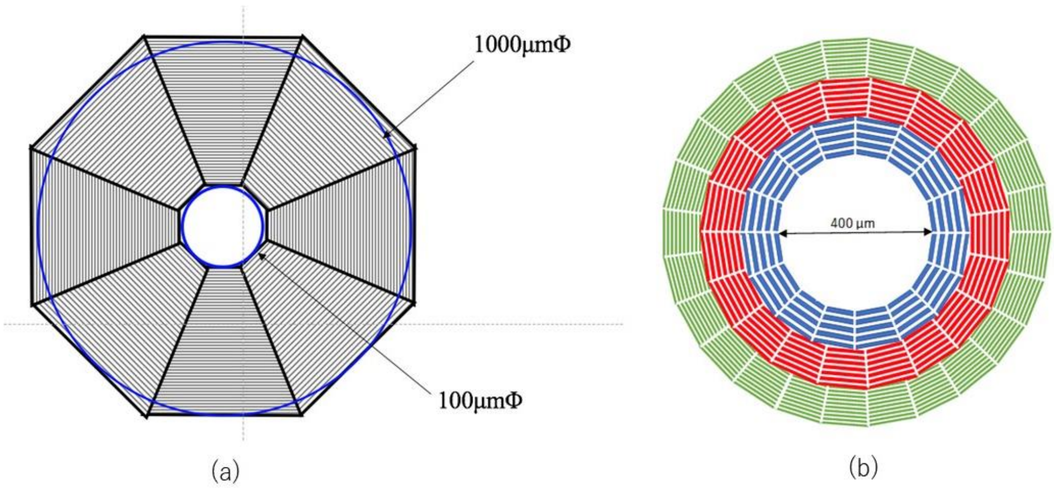


Fig. 3. Conceptual drawings of CZP. (a) Conventional CZP with eight segments (octagonal CZP, O-CZP) and (b) newly developed CZP with 60 segments (beam-shaping CZP, BS-CZP).

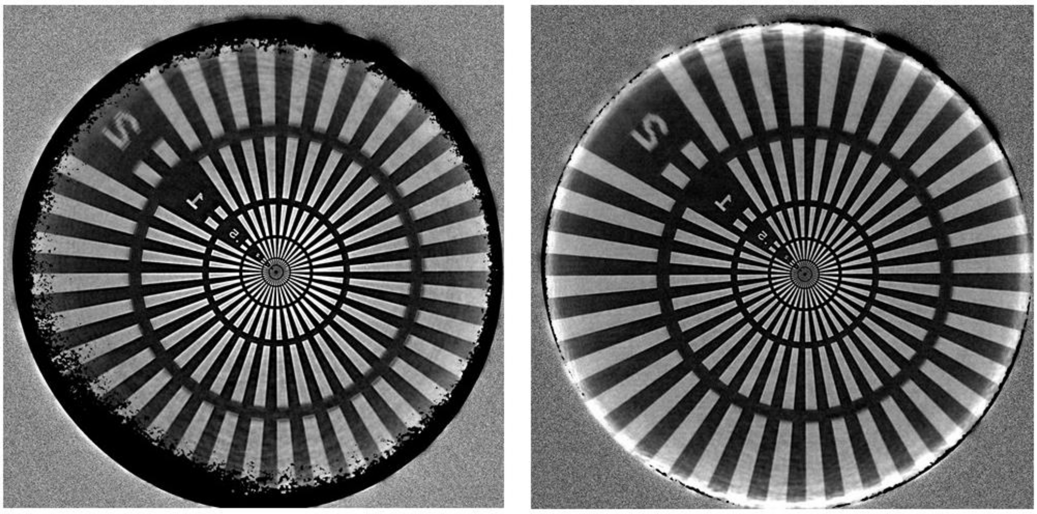

Fig. 4. X-ray images of the test chart (with flat-field correction). (left) with O-CZP and an exposure time of 10 s. Fringe noises are seen at the edge of patterns in the peripheral region of the field of view. (right) with BS-CZP and an exposure time of 2 s. Fringe noises are rarely seen.

multiple diffraction gratings with an equally spaced pitch (Fig. 3). Since the diffracted beam from each segment overlaps at the object plane, the intensity of the illuminating beam at the object plane is proportional to the number of segments of the CZP (in the case of incoherent illumination). Conventional CZP (octagonal CZP, O-CZP) consists of eight segments (Fig. 3a) [3], whereas the newly developed CZP (beam-shaping CZP, BS-CZP) has 60 segments (Fig. 3b) [4]. Therefore, the illumination intensity could be 7.5 times higher than when using the conventional CZP.

Figure 4 shows X-ray images of a tantalum test chart (NTT-AT) obtained with O-CZP (left) and with BS-CZP (right). The X-ray energy is 8 keV. The intensity of the image is approximately four times higher with BS-CZP. Moreover, the fringe noise and edge-enhanced contrast observed in Fig. 4 (left) are rarely seen in Fig. 4 (right). Hence, employing BS-CZP improves both the imaging properties and illumination intensity.

Yasumasa Takagi[*1], Akira Yasui[*1], Akihisa Takeuchi[*2], and Kentaro Uesugi[*2]

[*1] Spectroscopic Analysis Group II, Spectroscopy and Imaging Division, Center for Synchrotron Radiation Research, JASRI

[*2] Imaging Group, Center for Synchrotron Radiation Research, JASRI

**References:**

[1] E. Ikenaga et al., *J. Electron Spectrosc. and Relat. Phenom.* **190**, 180-187 (2013).

[2] E. Ikenaga et al., *Synchrotron Radiation News* **31**, 10 (2018).

[3] A. Takeuchi et al., *J. Phys. Conf. Ser.* **186**, 012020 (2009).

[4] U. Vogt et al., *Optics Lett.* **31**, 1465 (2006).