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 experimental tables for X-ray nano-CT, while EH2 contains HAXPES and micro-CT. In FY2020, an interlock system with a fast-closing shutter was installed for HAXPES system, and high-speed imaging system was also installed. The details are described herein.

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 allow in situ HAXPES measurements for liquid and gaseous samples which held in an atmospheric environment cell. This technique is currently under development in collaboration with a Partner User. However, accidental liquid scattering from the environment cell may cause the failure of the analyzer during the measurement. To make the equipment available for public use in the future, it is necessary to improve its safety against accidents. Therefore, an interlock system with a fast-closing shutter was installed to prevent the failure of the analyzer in FY2020.

In the environmental cell, the liquid sample inside is sealed by a SiN thin film of several tens of nanometers in thickness. When the SiN film is broken during the HAXPES measurement of a liquid sample, the liquid in the environmental cell leaks into the measurement chamber, causing the vacuum deterioration and contamination of the analyzer. To prevent the analyzer from discharging due to vacuum deterioration, we have already introduced an interlock system that works with a vacuum gauge to shut off the high voltage applied to the electrodes of the analyzer when the vacuum exceeds a threshold value. Since the introduction of this system, no failure of the analyzer caused by discharging due to vacuum deterioration has occurred. On the other hand, scattered liquid leaking into the analyzer can cause insulation breakdown due to electrode contamination. Therefore, an interlock system was introduced to instantly close a shutter between the environment cell and the entrance of the analyzer to prevent contamination by the scattered liquid.

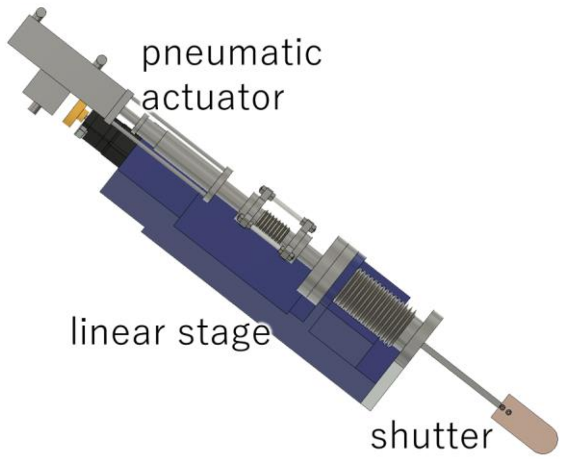

Fig. 1. Schematic of the shutter.

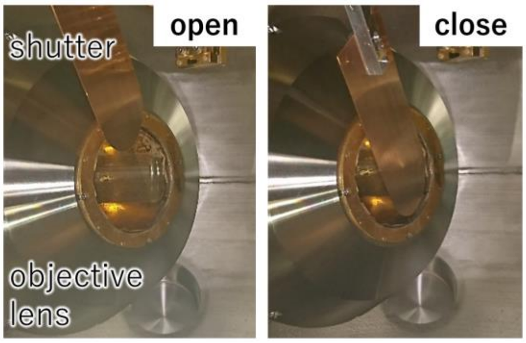

Fig. 2. Shutter in open and closed states.

A schematic of the shutter is shown in Fig. 1. The copper plate at the tip has a thickness of 0.3 mm. It is operated by a pneumatic actuator with a stroke of 30 mm using a solenoid valve. It is mounted on a 120 mm linear stage and can be retracted when the interlocking system is not needed for measurement. Figure 2 shows the open and closed states of the shutter. When open, the shutter is in a position that does not interfere with the detection of photoelectrons, and when closed by the interlock, it covers the entrance of the analyzer.

The breakage of the photoelectron transmission window is detected by the deterioration of the vacuum. When the vacuum exceeds 5×10^{-4}Pa, the relay of the controller is activated and the shutter closes within one second. Although the entry of small amounts of scattered liquid into the analyzer cannot be completely prevented, the contamination of the electrodes in the analyzer can be considerably reduced. This interlock system prevents the failure of the HAXPES apparatus and will ensure the stability of HAXPES experiments after the environmental cell becomes available for public use in the future.

3. Installation of high-speed measurement system

A high-speed measurement system has been installed at BL47XU. The system consists of a high-speed X-ray shutter made of a Ta plate on a galvanometer scanner, a high-speed X-ray imaging detector (Fig. 3), and a delay pulse generator (T3965-02, TSUJICON). An example of the system connection is shown in Fig. 3. The shutter, detector, and external field generator for the sample are

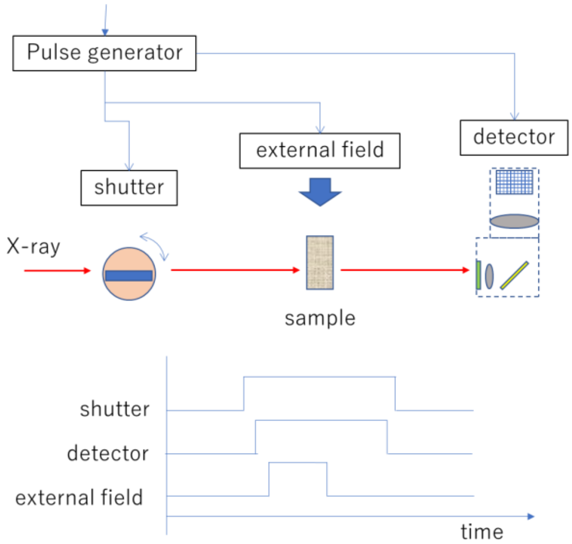

Fig. 3. Example of system connection (upper) and timing chart for measurements (lower). Each plateau position shows open, recording, and forcing of activating a sort of external field in the case of the demonstration (Fig. 5).

controlled by TTL pulses from the pulse generator.

The delay pulse generator has one channel for input to the external trigger, four channels for output, and a temporal accuracy of 10 ns. This is sufficient for controlling most high-speed imaging experiments. The shutter covers the size of a 1-mm-square X-ray beam. The time duration for opening and closing is each approximately 1 ms. There are some devices for applying an external field to samples, such as an overcurrent generator, a laser heating system, and tensile and compression devices. It is easy to handle such devices as they can be programed to start a sequence with a TTL signal.

The high-speed X-ray image detector consists of a scintillator screen, an objective lens for visible light, and a high-speed camera. The scintillator is a LuAG ($Lu_3Al_5O_{12}:Ce^+$) single crystal with a thickness of 500 μm. This might seem to be very thick for high-spatial-resolution imaging, but it is highly suitable for high-speed imaging. The high-speed camera is SA-Z (Photron [3]). It achieves a frame rate of 20 kHz with a 1024 x 1024 pixel format. The ADC is 12 bits. If we reduce the number of pixels to, for example, 640 x 280, it achieves a frame rate of 100 kHz. The effective pixel size is 1.1 um/pixel.

The X-ray flux density and efficiency of the detector depend on the X-ray energy. Therefore, the relationship between energy and possible frame rates was evaluated. As a result of the evaluation, more than 2000 ADCs were obtained on the high-

Fig. 4. High-speed X-ray image detector at experimental hutch 1.

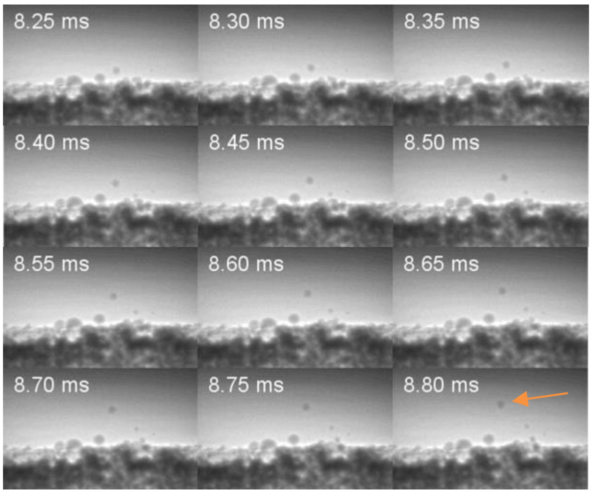

Fig. 5. Test measurement images of high-speed imaging. The field of view of each image is 240 um x 150 um, which has been cropped from the original image. Metal powders were heated with a CO_2 laser. The velocity of the particle (~ ϕ10 um) indicated by the orange arrow was estimated to be 70.4 mm/s. The images were provided by Assoc. Prof. Morishita (Kyushu Univ.).

speed camera (20 kHz mode) in the energy range between 15 keV and 37.7 keV.

As a demonstration of the system, the movement of metal particles during flash heating was recorded (Fig 5). The CO_2 laser was irradiated on the metal particles for a few hundred milliseconds during the recording of the X-ray image. The frame rate was 20 kHz and the field of view (beam size) was 800 µm square. The effective pixel size was 1.1 µm/pixel. The X-ray energy was 20 keV. The movie clearly shows how the gas inside expands as a result of heating and pushes out the particles near the surface layer. It was also confirmed that some of the particles had been pushed back by the laser pressure or gravity.

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[3] https://www.photron.co.jp/