

## BL46XU (Engineering Science Research III)

### 1. Introduction

BL46XU is an industrial application beamline with an undulator light source to promote the utilization of synchrotron radiation by industry. A multi-axis X-ray diffractometer is installed in the first experimental hutch (EH 1). In addition, X-ray imaging and micro-focus X-ray diffraction are available in the open space of EH 1. In the second experimental hutch (EH 2), two hard X-ray photoelectron spectroscopy (HAXPES) systems are installed. In FY2018, for a multi-axis X-ray diffractometer, a new point detector, and signal processing devices were installed to realize a high counting rate. For HAXPES, a new electron energy analyzer was installed to achieve more stable and reliable measurements.

### 2. Optics and Performance

The light source is a standard in-vacuum undulator at SPring-8, and a liquid nitrogen cooled Si (111) double-crystal monochromator is adopted for the optics. The tunable energy range is 6–35 keV. Two Rh-evaporated mirrors (70-cm length, horizontal reflection direction) are placed in the most downstream part of the optics hutch to eliminate

harmonics. The mirrors are curved for a horizontal light focus. A Si (111) channel-cut monochromator is placed between the monochromator and the mirrors to achieve incident X-rays with fine energy resolution. Figure 1 shows the beamline layout of BL46XU.

### 3. New equipment and development

#### 3-1. New point detectors and signal processing devices for the HUBER multi-axis X-ray diffractometer

On the upstream side of EH 1, a HUBER multi-axis X-ray diffractometer was installed. This diffractometer can perform not only general X-ray diffraction/scattering measurements but also various types of diffraction/scattering experiments. For example, high angular resolution diffraction, residual strain measurements, anomalous X-ray scattering, grazing-incident X-ray diffraction (GIXD), X-ray reflectivity, micro-beam diffraction and various *in situ* measurements can be performed. To carry out X-ray diffraction/scattering measurements, a point, line, or two-dimensional (2D) detector can be attached to the detector arm of this diffractometer.

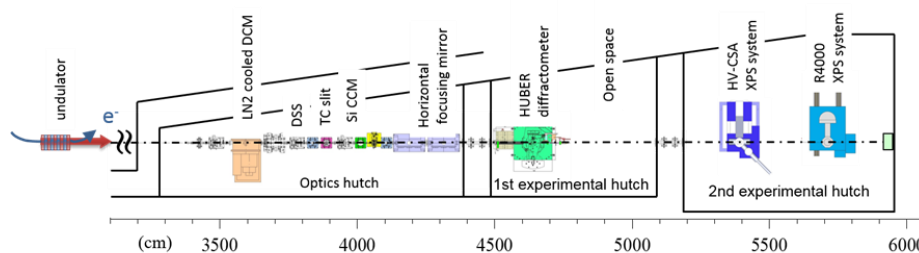


Fig. 1. Beamline layout of BL46XU.

In FY2018, new point detectors (Scintillation Detector, C30LaBr25B (LaBr<sub>3</sub>:Ce<sup>3+</sup>); FMB Oxford) and signal processing devices (Pre-Amplifier, CP10-A and Pulse Processor, C400; FMB Oxford) were introduced to enhance the counting rates. Because the decay time of the LaBr<sub>3</sub>:Ce<sup>3+</sup> scintillator is 16 ns<sup>[1]</sup>, which is much shorter than that of NaI:Tl, wide dynamic range measurements with a high counting rate can be realized. Figure 2 shows the newly introduced scintillation detector, pre-amplifier, and pulse processor. The X-rays diffracted by the sample pass through slits, attenuator, and vacuum paths. Then they are detected by the scintillation detector.

In an experiment to measure the dead-time characteristics of the scintillation detector, the X-ray intensity diffracted by a CeO<sub>2</sub> powder sample (SRM674b; NIST) was recorded as a function of the incident X-ray intensity. The X-ray intensity was varied by changing the number of attenuators located upstream of the ion chamber. The blue points in Fig. 3 show the output count rate as a function of the input count rate. Table 1 summarizes the measurement conditions. The output count is

linear to the input count below approximately  $3.0 \times 10^5$  cps, but above this value the dead-time error is observed. Fitting a non-paralyzable dead-time model of equation

$$N_{\text{meas}} = N_0 / (1 + N_0 \cdot \tau)$$

to the measured counts  $N_{\text{meas}}$  yields a characteristic dead-time  $\tau = 65$  ns. The red points in Fig. 3 are the corrected values with the dead-time correction using a dead-time of 65 ns. Performing the dead-time correction indicates that the linear

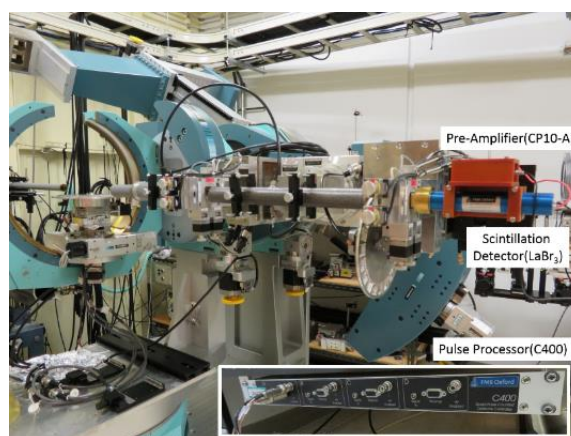


Fig. 2. LaBr<sub>3</sub> (Ce) scintillation detector, pre-amplifier (CP10-A) attached to the detector arm, and pulse processor (C400).

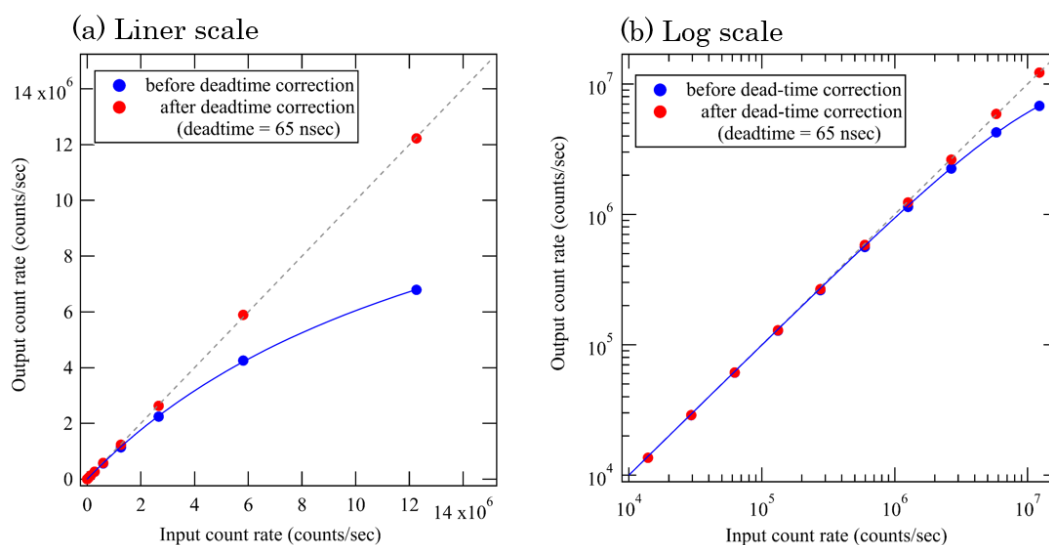


Fig. 3. Typical count rate characteristic displayed on (a) a linear scale and (b) a log scale.

Table 1. Measurement conditions for the dead-time characteristics.

X-ray energy	12.4 keV
Bunch mode	B mode (4-bunch train $\times$ 84)
Input X-ray	CeO <sub>2</sub> 111 reflection
High voltage	-1200 V
Discriminator range	0.07-1.5 V
Pulse counter	CT08 (Tsuji Electronics Co., Ltd)

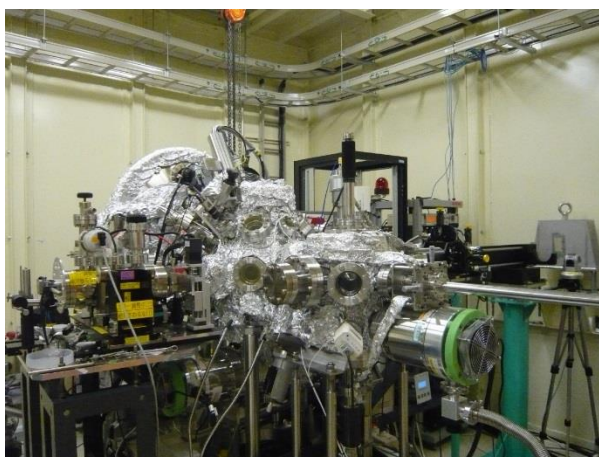


Fig. 4. Photograph of the HAXPES system in the second experimental hatch of BL46XU.

region is expanded to approximately  $1.0 \times 10^7$  cps. This scintillation detector is used for X-ray reflectivity, GIXD, etc.

### 3-2. New electron energy analyzer for HAXPES

In 2008, a HAXPES measurement system equipped with an electron energy analyzer was installed. This system is open to industrial researchers and provides a powerful tool to directly explore electronic structures deep inside a material such as electrode/dielectric interfaces buried in gate stack structures, which are not accessible by conventional XPS [2]. However, aging equipment resulted in unstable and unreliable measurements due to the leakage current at the pre-retarding lens part. To

address these critical issues, we developed a new electron energy analyzer (R4000L1-10kV; Scientaomicron) for the HAXPES measurement system (Fig. 4). This system can analyze electron kinetic energies up to 10 keV. The R4000L1-10kV analyzer consists of a hemispherical analyzer, pre-retarding lens system, 2D event-counting detector equipped with a multi-channel plate, phosphor screen, and charge-coupled device camera. The entrance slit of the analyzer has a rectangular shape with several selectable sizes in the energy-dispersive direction and a fixed size (30 mm) in the non-dispersive direction. The lens mode can be in the transmission mode or angular mode. The pass energies are changeable from 5 eV to 500 eV. Currently, the upgraded HAXPES system provides stable operations for user experiments.

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### References:

- [1] <https://www.fmb-oxford.com/products/detectors-diagnostics/fmb-scintillation-detectors/>
- [2] H. Oji et al., *J. Surf. Anal.*, **21**, 121(2015).