5-1. Public Beamlines

BL01B1
XAFS

1. Introduction

BL01B1 is a public beamline dedicated to X-ray Absorption Fine Structure (XAFS) measurements using X-rays over a wide energy range between 3.8 keV and 113 keV. It is used for various applications in materials science and chemistry. In FY2019, BL01B1 beamline and its experimental station operated stably for user research. The latest beamline information is available on the website at https://bl01b1.spring8.or.jp/, including the performances of the XAFS spectrometer and other equipment as well as the appropriate user manuals. This report describes the improvements at BL01B1 in FY2019.

2. New current amplifier with an external low-pass filter

The current amplifier is indispensable in XAFS measurements. Replacing it with a new one that has a higher specification is crucial because the manufacturer discontinued the current model. Based on a test use performed last year, current amplifier CA5350 (NF Corporation) was chosen as a replacement candidate. However, when CA5350 was used in several bunch modes of the storage-ring, it became saturated, depending on the filling pattern, because it has a wide bandwidth of 500 kHz. This wide bandwidth causes nonlinearity of the beam intensity measurements. Unfortunately, the filter equipped as a default function of CA5350 cannot eliminate it because it only smooths the signal at the output stage. Thus, an external filter was developed to smooth the signal structure at the input stage. The optimum filter configuration was examined via a SPICE simulation for a circuit, which included the input stage of the publicly available high-speed

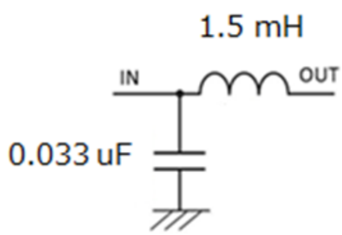

Fig. 1. Circuitry diagram of the L-type LPF.

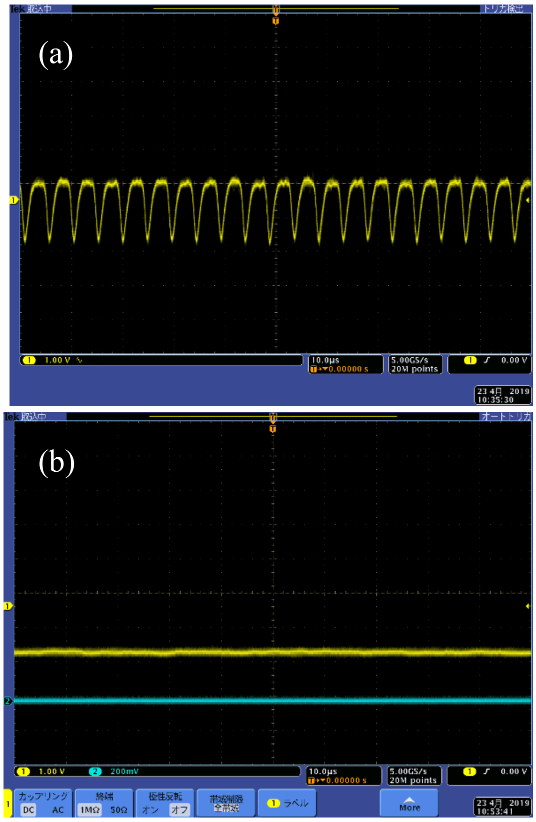

Fig. 2. Waveforms of the I0 signal (a) without and (b) with filter.

current amplifier of NF Corporation. The L-type LPF filter is suitable to resolve the saturation issue (Fig. 1).

Figure 2 show the impacts of the filter on the waveform of the output of the I0 ion chamber in the D-mode several bunch operation at BL01B1 viewed with CA5350. In the D-mode operation, the signal of the ionization chamber contains a large AC component because the full-fill part and the isolated bunch part are arranged asymmetrically. Without the filter, the structure with an amplitude of about 1 V from the frequency of 208 kHz is visible due to the asymmetric filling (Fig. 2(a)). However, after passing through the filter, the structure at 208 kHz almost disappears (Fig. 2(b)).

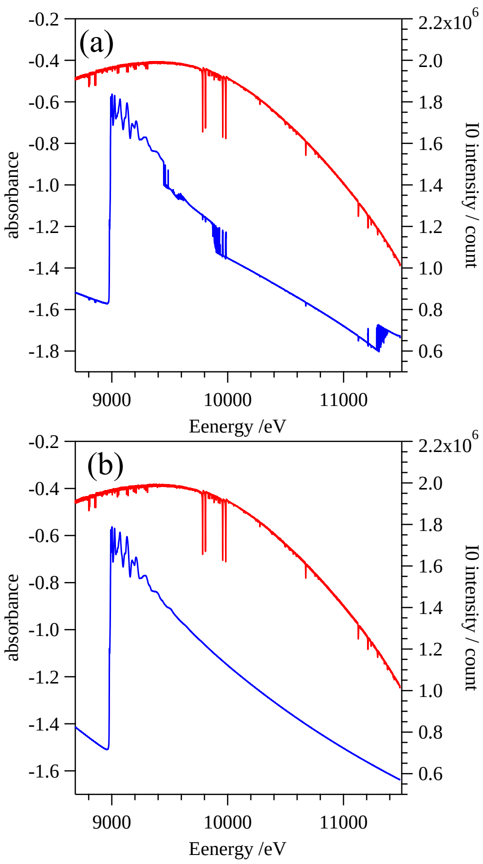

Fig. 3. Cu K-edge QXAFS spectrum of the Cu foil (blue line) and I0 spectrum (red line) measured (a) without and (b) with a filter.

The filter was used for XAFS measurement with the CA5350 under the storage ring operation in D-mode. Figure 3 show Cu K-edge QXAFS spectrum of the Cu foil (blue line) and I0 spectrum (red line) measured (a) without and (b) with a filter. It can be seen that the discontinuity appears in the middle of the spectrum measurement without the filter (Fig.3(a)). It was caused by the saturation of I0. On the contrary, smooth spectrum was obtained with the filter (Fig.3(b)).

3. Simultaneous measurements of XAFS and XRD

To meet user demand, we developed a system to perform XAFS and XRD measurements simultaneously under an *in situ* condition in FY2014. This simultaneous measurement system provides information about crystal structures, electronic states, and local structures of materials under the same conditions. Combining these data provides precise information about the chemical and structural changes in materials during chemical reactions and synthetic processes. This simultaneous measurement system also improves the measurement reliability about the sample position, sample sameness, and measurement conditions. Until FY2017, the XAFS and XRD simultaneous measurement metal cell was developed and improved [1-3]. Temperature and gas ambient controls were realized for the test measurements. The achieving temperature in this measurement was 923 K, and the temperature was unstable in the high-temperature region (873–923 K). However, the target temperature (1073 K) was not achieved.

To reach the target temperature and realize stable temperature control, a new cell was developed,

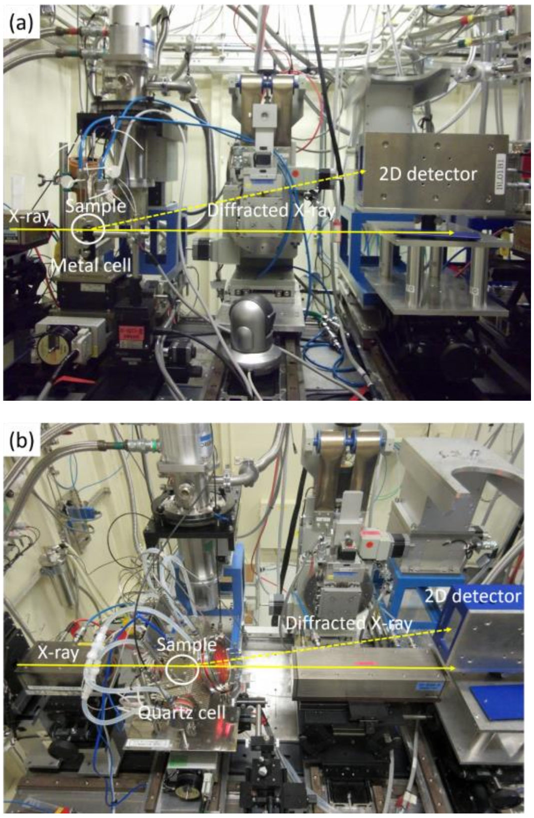

Fig. 4. XAFS-XRD simultaneous measurement system: (a) previous metal cell and (b) new quartz cell.

where the body was made of quartz. Figure 4 shows the simultaneous measurement system of (a) the previous metal cell and (b) the new quartz cell. The heating system is limited to around the sample, and the sample is covered in a tube furnace. The tube furnace maintains a stable sample temperature and can easily reach a temperature up to 1123 K (Fig. 5). The overshoot to set the temperature is within 10 K when the heating rate is 10 K/min. The sample holder for placing the sample in the new cell is similar to that for the *in situ* cell at BL01B1. The water flow system cools the incident window and exit window. When the sample temperature is around 1073 K, the surface temperatures of the windows are below 353 K. The gas flow and water cooling systems of the new quartz cell are similar to the *in situ* cell existing at BL01B1. Hence, these systems can be easily applied to the new cell. Because the height of the cell was designed with respect to the incident X-rays, the sample position can easily be located. The sequence of XAFS and XRD simultaneous measurements is not changed. The details are described elsewhere [2]. In FY2020, the new cell will be available for public use. Users' feedback will be used to improve the new cell.

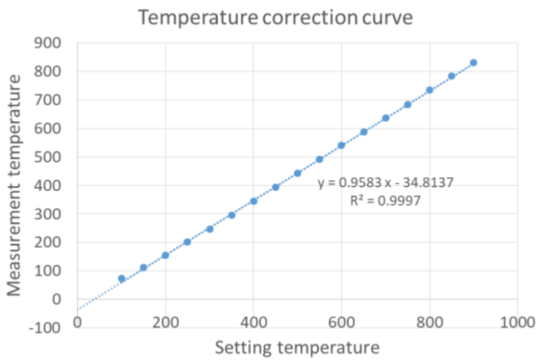

Fig. 5. Temperature correction curve for the new quartz cell.

Kazuo Kato[*1], Toshiaki Ina[*1], Tomoya Uruga[*1], and Togo Kudo[*2]

[*1] Spectroscopy and Imaging Division, Center for Synchrotron Radiation Research, JASRI

[*2] Information-Technology Promotion Division, JASRI

References:

[1] *SPring-8/SACLA Annual Report*, 2014, p33.

[2] *SPring-8/SACLA Annual Report*, 2015, p27.

[3] *SPring-8/SACLA Annual Report*, 2017, p27.