5-1. Public Beamlines

BL01B1
(XAFS)

1. Introduction

BL01B1 is a public beamline for XAFS (X-ray Absorption Fine Structure) measurements using X-rays in a wide energy range between 3.8 keV and 113 keV. It is used for various applications in materials science and chemistry. In FY2018, BL01B1 beamline and its experimental station stably operated for user research. The latest beamline information is on the website at https://bl01b1.spring8.or.jp/, including the performances of the XAFS spectrometer and equipment as well as user manuals. This report describes the improvements at BL01B1 in FY2018.

2. Simultaneous measurement of XAFS and IR

To meet user demand, we developed systems to perform XAFS and other analysis measurements simultaneously under an *in situ* or *operando* condition with the objective of investigating the change in local structures in materials during chemical reactions and synthetic processes. In particular, the XAFS-IR simultaneous measurement provides information about surface states, adsorbed species, electronic states, and the local structures of materials under the same conditions [1]. The simultaneous measurement system was installed and tested in FY2017, but was improved in FY2018. One of the improvements in FY2018 enhanced the air purging capability in the light path between the IR spectrometer and the detector. For IR measurements, the light path needs to be in a vacuum or filled with nitrogen gas because atmospheric carbon dioxide and water vapor absorb IR lights and create background absorption peaks in the IR spectra. In the XAFS-IR simultaneous measurement system, the IR light path is filled with nitrogen gas using an air purge system, which consists of acrylic boxes, pipes, and a nitrogen gas generator (Fig. 1(a)). Although the initial installation in FY2017 did not work well, improvements to the acrylic box and the optimization of the nitrogen gas supply, which both occurred in FY2018, led to the successful removal of carbon dioxide and water vapor in the IR light path. Figure 2 shows background spectra measured before and after the improvement, demonstrating that the absorption of carbon dioxide and water vapor are largely decreased by the improvement.

The improvement in FY2018 makes the XAFS-IR simultaneous measurement system more stable, leading to a shorter recovery time of the absorption intensity after a sample exchange. In addition, the sample reactor setting is simple in the acrylic chamber. Hence, sample exchange is easier.

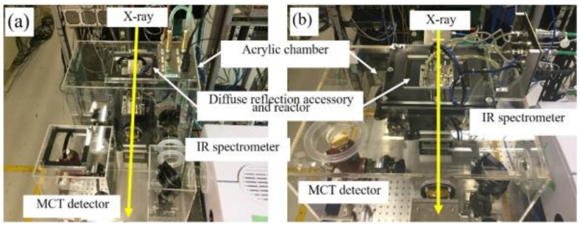

Fig. 1. XAFS-IR simultaneous measurement system: (a) before and (b) after improvement of acrylic chamber.

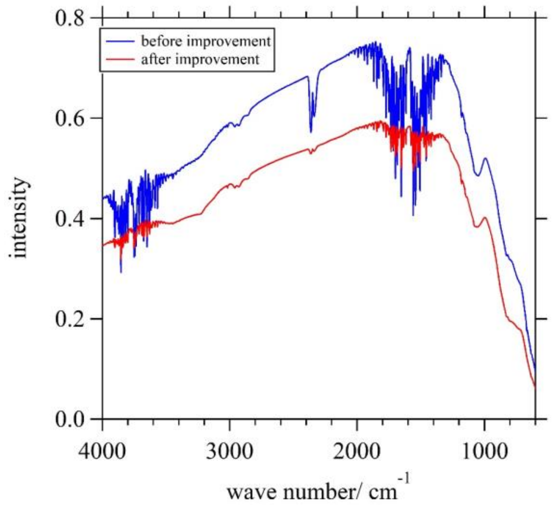

Fig. 2. IR spectra of the background measurement.

3. Evaluation of the current amplifiers for XAFS measurements

Because KEITHEKEY 428 was discontinued, the performance of the fast-current amplifiers, CA5350 (NF co.) and DLPCA-200 (FEMTO Messtechnik GmbH), were evaluated for XAFS measurements, to replace KEITHEKEY 428 used in SPring-8. The uniformity of the current amplifier's response time is essential, particularly in quick XAFS (QXAFS) measurements.

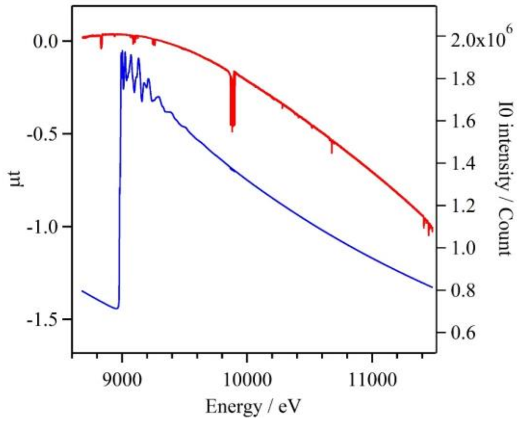

Fig. 3. Cu K-edge QXAFS spectrum of the Cu foil (blue line) and I0 spectrum (red line) measured using NF CA5350.

Figure 3 shows the Cu K-edge (8.9 keV) QXAFS spectra of a Cu foil together with the I0 spectra, measured using NF CA5350. The data accumulation time is 14.18 ms at each measurement point. Glitches occur around 9.9 keV when the filter is off for CA5350 or the operation mode is High speed for FEMTO. Figure 4 shows the XAFS spectra around 9.9 keV to highlight the structures of the glitches. In this situation, the input current pulse height exceeds the maximum input range of 10 V for NF, and the output is saturated for FEMTO (Fig. 5). To solve this problem, an external low-pass filter, which does not affect the input charge amount into the amplifier, is being developed.

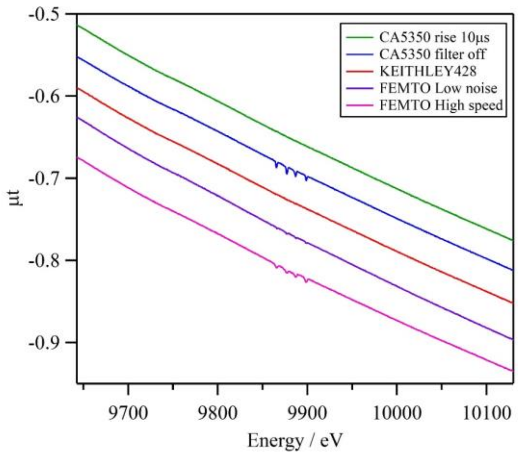

Fig. 4. Expanded XAFS spectra around 9.9 keV structure.

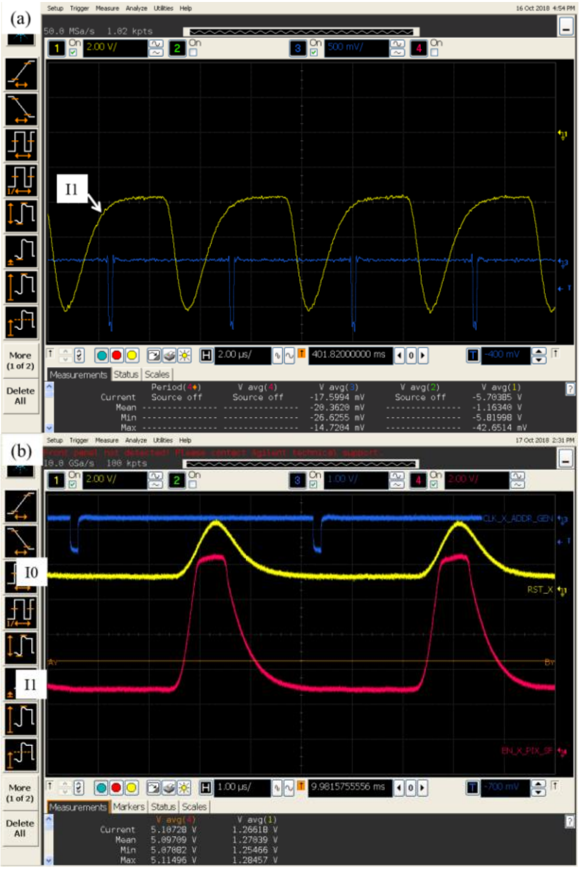

Fig. 5. Waveforms of (a) I1 (Yellow) Amp. Out signal of NF CA5350 and (b) I0 (Yellow) and I1 (Red) Amp. Out signals of FEMTO DLPCA-200. The blue lines of (a) and (b) are the trigger signals synchronized with each revolution of the electron bunch.

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Reference:

[1] SPring-8/SACLA Annual Report, 2017, p26.