5-1. Public Beamlines

BL01B1
XAFS I

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 recent years, a gas facility for in-situ/operando measurements using reactive gases has been improved [1], and a combined measurement system with other analytical methods such as X-ray diffraction and infrared spectroscopy has been developed and used by a lot of users [2]. In FY2022, the 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 specifications of beamline components and XAFS measurement systems as well as the appropriate user manuals. In this report, the improvements made to the fluorescence XAFS measurement system and the gas analysis system in FY2022 are described.

2. Improvement of fluorescence XAFS measurement system

The fluorescence XAFS measurement with a 19-element SSD is the most important measurement technique for dilute and thin-film samples and is used by more than 50% of all users. The measurement system had used XIA's xMAP digital signal processor (DSP) as a data processing system since FY2006, which enabled fluorescence XAFS measurements with simple and quick adjustments [3]. However, in recent years, the number of malfunctioning signal input channels increased, hindering fluorescence measurements. The DSP was upgraded to enable the common management and control of measuring instruments with other spectroscopic beamlines, and the control software and parameters for the X-ray fluorescence measurement were adjusted and verified. The newly introduced DSP is the APN504 from Techno AP, which has four input terminals per module, so the data processing system consists of five modules (Fig. 1). The acquisition of measurement data and setting of control parameters are carried out via a 10 Gbps Ethernet interface. This interface is independent of the network to which other measuring instruments are connected to prevent communication interference from other instruments. The APN504 is equipped to transfer measurement data to a control PC at high speed, and currently, QXAFS measurements can be made at intervals of as short as 40 ms per measurement point.

Fig. 1. Photograph of the new data processing system consisting of five APN504 modules.

3. Improvement of gas analysis system

Simultaneous XAFS and gas analyses of functional materials such as catalysts under a reactive gas atmosphere allow the observation of dynamic changes in the reaction properties, electronic state, and structure of the sample under the same reaction conditions. Furthermore, the reliability of measurement data is improved by eliminating the need to account for subtle differences in measurement conditions between individual analyses. A reaction gas control and analysis system that can be synchronized with XAFS measurements was constructed in FY2016 [4] and has been used for many operando XAFS measurements. In FY2022, a new gas analysis system was constructed to improve the performance and convenience of gas analysis (Fig. 2). The analysis system consists of high-speed micro–gas chromatography (μGC) for the rapid determination of the reactor outlet gas and quadrupole mass spectroscopy (QMS) for the real-time monitoring of gas composition. The μGC device is Agilent 990 (Agilent Technologies, Inc.) equipped with three different analytical units, allowing quantitative analysis of hydrogen gas, which was not possible with the previous analysis system. The QMS device is GSD350 OmniStar (Pfeiffer Vacuum GmbH), which is capable of analyzing gases with mass numbers up to 200. It is resistant to corrosive gas, which enable the measurement of corrosive gases such as NH_3 and NO. These analyzers and He and Ar gas cylinders for supplying carrier gases to μGC are housed in a movable cabinet for easy installation in the experimental hutch. The exhaust gases from the reactor can be simultaneously analyzed qualitatively and quantitatively by connecting the gas inlet port of this cabinet to the reactor outlet through a single pipe. This system is equipped with an uninterruptible power supply (UPS) that allows the system to be brought into the experimental hutch without disconnecting the power supply to the system after the system has been started up and adjusted outside the experimental hutch.

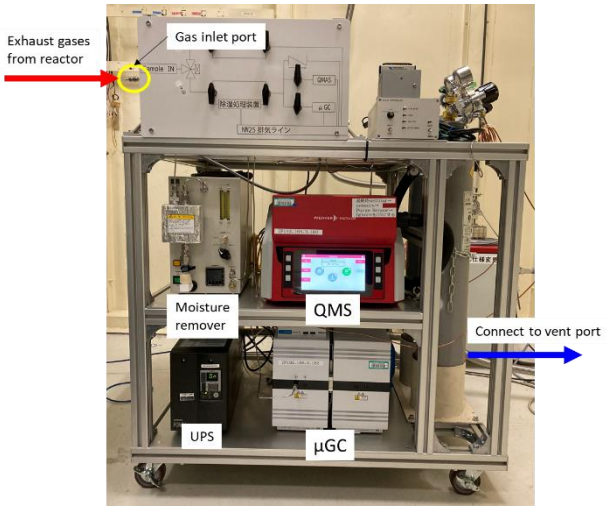

Fig. 2. Photograph of gas analysis system housed in a movable cabinet.

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