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 FY2021, 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 specification of beamline components and XAFS measurement systems as well as the appropriate user manuals. This report describes the improvements of the gas handling system at BL01B1 in FY2021.

2. Improvement of gas handling system for in situ XAFS measurement

XAFS is one of the most suitable analytical methods for in situ observation because it can be applicable under various sample environments such as gas, in solution, and at high temperatures. In in situ measurements of catalysts and electrochemistry, the use of reactive gases has already become commonplace, and a gas handling system to safely supply and exhaust reactant gases is an important and necessary experimental component.

The gas handling system at BL01B1 has been contributing to the safe and efficient performance of these experiments since 2006 [1]. Recently, however, it has become necessary to be able to use many gas species simultaneously in order to conduct experiments under more real reaction conditions. Therefore, the gas handling system was updated to allow more gas species to be used simultaneously in FY2021.

Figure 1 shows a schematic diagram of the gas handling system of BL01B1. Multiple reaction gases can be supplied to the measurement sample from cylinders stored in their respective cabinets, and the reaction gases used can be detoxified and released outdoors for safe handling. The reactant

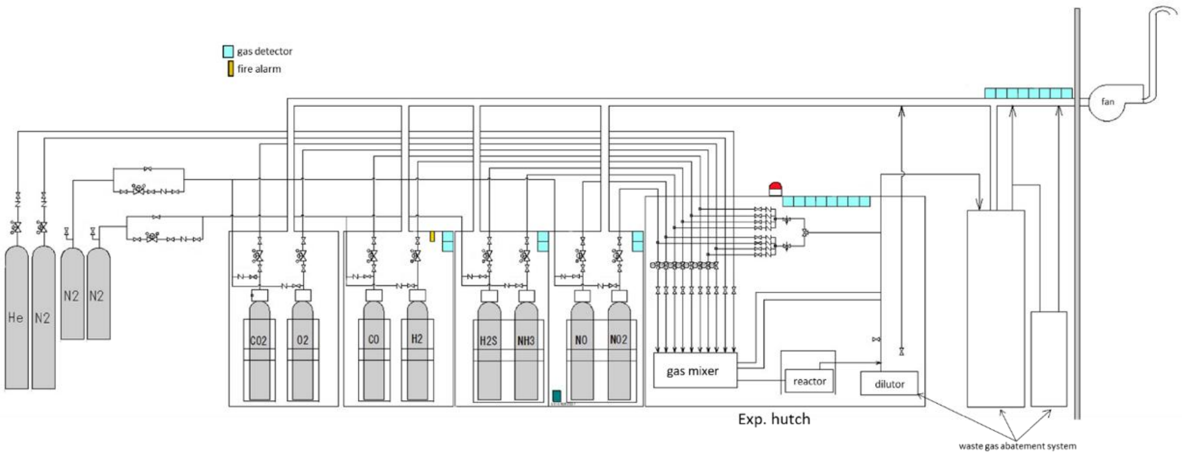

Fig. 1. Schematic of gas handling system of BL01B1.

gases that can be used in the system are H_2, CO, NH_3, hydrocarbons, NO, NO_2, O_2, and H_2S. This update includes the addition of a supply line for combustible and combustion-supporting gases and a cylinder cabinet for combustion-supporting gases (Fig. 2). Consequently, the number of reactant gas supply lines is four for each of the combustible and combustion-supporting gases.

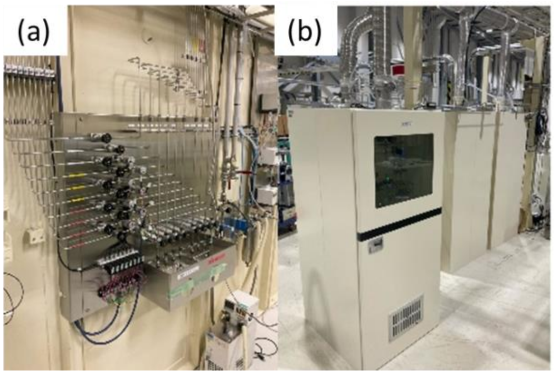

Fig. 2. Photographs of supplying piping for reactant gases in experimental hutch (a) and cylinder cabinet for combustion-supporting gases (b).

As part of the gas system improvements, the safety interlock system was also improved (Fig. 3). Four gas detectors were added to each of the three stations to accommodate the increased number of gases used simultaneously. In addition, the interface of the interlock control panel was changed from a combination of push buttons and indicator lamps to a touch panel to accommodate the increased number of gas leak monitoring points. Consequently, the operability of the interlock system and the visibility of alarm conditions were improved. Furthermore, the interlock system has the ability to remotely check alarms and release interlocks via LAN.

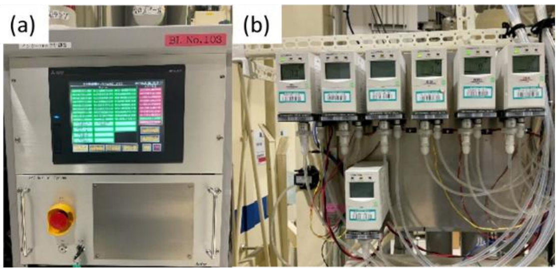

Fig. 3. Photographs of control panel (a) and gas detectors (b) of interlock system.

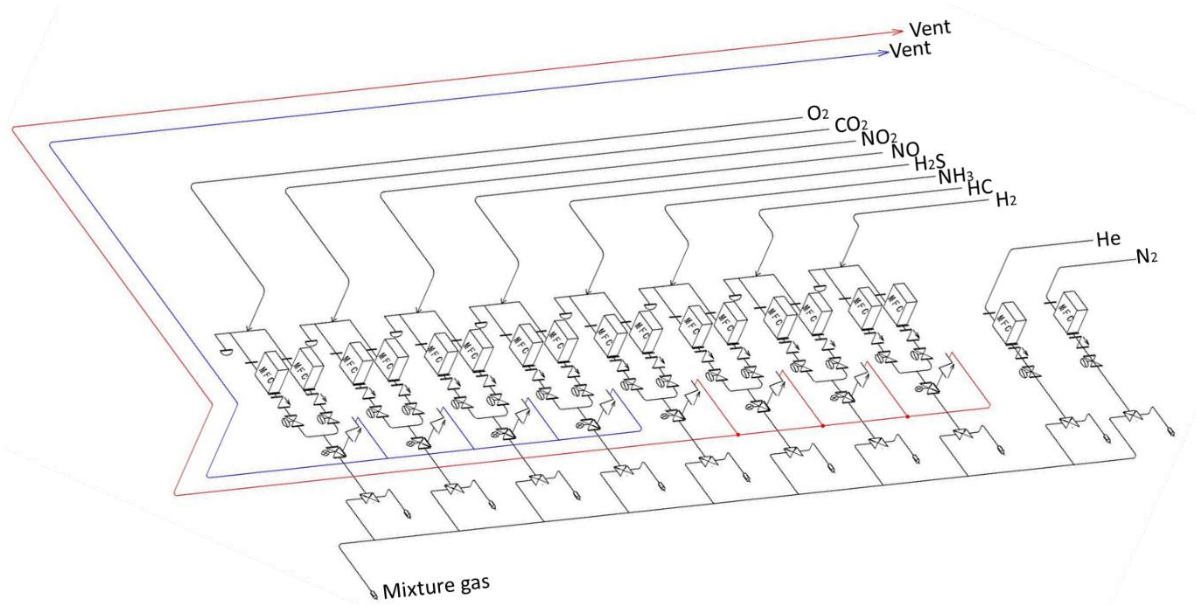

Fig. 4. Schematic of gas mixing system of BL01B1.

In this update, a new gas mixing system was also developed to generate a mixture of two or more gases, which adjusts the flow rate of eight reactant gases and He and/or N_2 gas. Figure 4 shows the schematic diagram of the gas mixing system and that in the place on the experimental hutch is shown in Fig. 5(a). The mixing system consists of mass flow controllers for controlling the flow rate of gas, pneumatically operated stop valves, and three-way switching valves. The flow rate of the reactant gas can be adjusted in the range from 0.2 ccm to 310 ccm with two mass flow controllers with full scales of 10 ccm and 300 ccm, allowing a wide concentration range to be set with high accuracy. Furthermore, because this system can also supply independent flow-regulated gases, more gas conditions can be generated by combining it with other gas equipment. The system is controlled from the LabVIEW program on a PC via socket communication over LAN (Fig. 5(b)). In the future, we plan to construct a system to synchronize it with XAFS measurement system, gas analyzer, and furnace controller. This synchronization system will allow us to perform the in situ measurement under a precisely controlled sample environment.

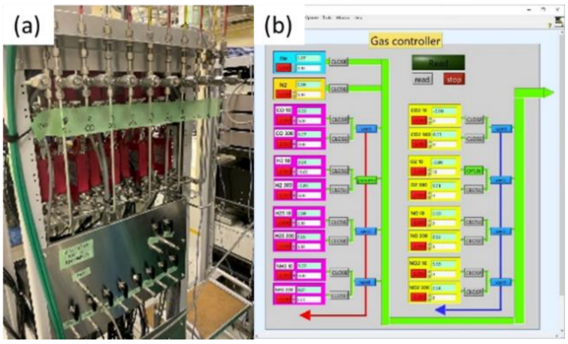

Fig. 5. Photographs of gas mixing system in place on experimental hutch (a) and control program built in LabVIEW (b).

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Reference:

[1] Tanida, H. Kato, K. & Uruga, T. (2020). *SPring-8 Annual Report FY2006*, 81–82.