BL14B2
(Engineering Science Research II)

1. Introduction

BL14B2 at SPring-8 is a bending magnet beamline designed for engineering science research via the X-ray absorption spectroscopy (XAS) measurements. We have developed various measurement systems to realize easy and high-throughput operations of XAS measurements. In FY2018, we improved the gas distribution and exhaust gas treatment systems to increase the gas pressure. We also modified the automatic systems for high-throughput XAS measurements at low temperatures.

2. Remodeling of the gas distribution and exhaust gas treatment systems to increase the gas pressure

The number of engineers conducting *in situ* XAS measurements while synthesizing catalysts or monitoring catalytic reactions is increasing. Since many of these experiments are chemical reactions in a reactive gas atmosphere, the use of gas distribution and exhaust gas treatment systems is essential for safety. When gas equipment was introduced at the initial stage at BL14B2, it was assumed that experiments were performed under pressure conditions close to atmospheric pressure, such as *in situ* measurements of catalysts for automobile exhaust gas [1]. For this reason, systems were introduced that can adjust the working pressure in a range from normal pressure to about 0.2 MPa.

Previously, most *in situ* measurements were under the reaction conditions of catalysts for automobile exhaust gas; however, in recent years, the number of *in situ* measurements under catalytic reactions to elucidate the reaction mechanism of organic synthesis has increased. Many organic synthesis reactions with practical use proceed under high pressure. Hence, structural analysis of catalytic active sites under a pressurized gas atmosphere is demanded. Therefore, the gas distribution and exhaust gas treatment systems were modified to conduct experiments under a pressurized gas atmosphere.

Since gas pressure supplied from the gas cylinder is about 0.3 MPa or less, the pressure regulators and pressure gauges in the gas lines other than hydrogen sulfide (H_2S) were replaced to supply gas at a pressure up to about 0.9 MPa. Figure 1 schematically depicts the gas distribution part. For the H_2S line, the necessary equipment is being evaluated. In addition, a mass flow controller that can adjust the gas flow rate under a pressurized gas atmosphere in the range of 0.5–0.8 MPa was added.

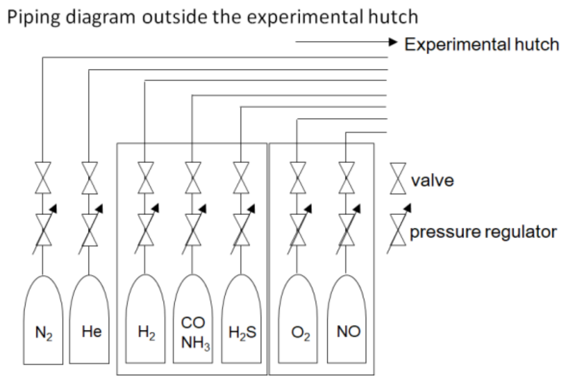

Fig. 1. Schematic diagram of gas distribution part.

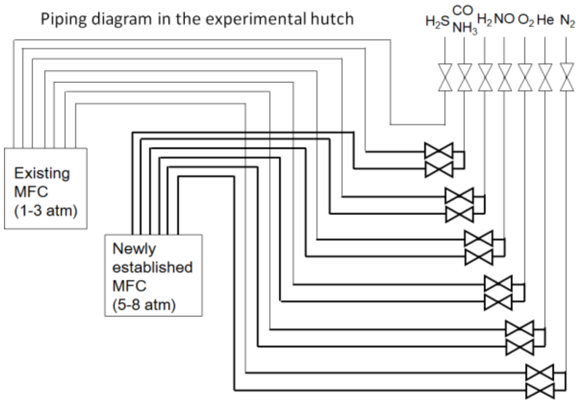

Fig. 2. Schematic diagram of mass flow controllers and gas piping in the experimental hutch.

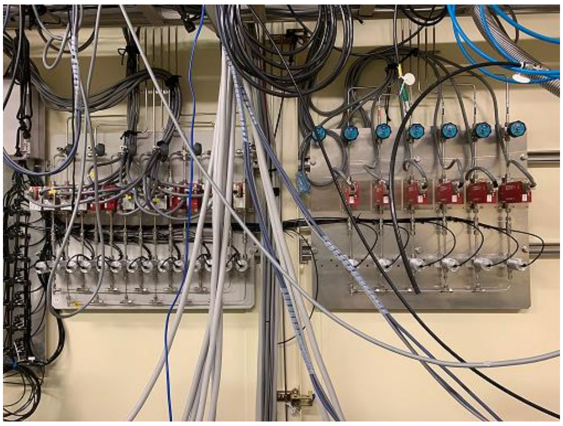

Fig. 3. Picture of mass flow controllers.

The gas piping was also modified. Figure 2 schematically diagrams the mass flow controllers and the gas piping in the experimental hutch. The piping was branched downstream of the supply valves for each gas type. The thick lines in Fig. 2 indicate newly added pipes and switches. Figure 3 shows a photograph of the mass flow controller. The devices on the left panel are the mass flow controllers capable of controlling the pressure near atmospheric pressure, while those on the right panel are the newly added mass flow controllers to adjust the flow rate under a pressurized gas atmosphere to 0.5–0.8 MPa.

In FY2019, the operation of gas pressure and flow rate control and a trial for *in situ* measurements of catalytic reactions will be conducted under a pressurized gas atmosphere. The system is slated for user experiments in FY2020.

3. Improvement of the automatic system for low temperature XAS measurements

The advantage of XAS measurements at low temperature is that damping of the EXAFS oscillation can be suppressed by decreasing Debye-Waller factor, improving the signal to noise (S/N) ratio in EXAFS oscillations. At BL14B2, a temperature-controllable cryostat to measure XAS at low temperature (10–300 K) was installed. We also developed an automatic system for low temperature XAS measurements. This system can realize automatic XAS measurements for up to 15 samples at 10 preset temperatures [2]. To improve the accessibility and efficiency of XAS measurements at low temperature, we modified the automatic XAS measurement program at low temperature.

Fig. 4. Front panel of the automatic XAS measurement program.

Figure 4 shows the front panel of automatic XAS measurement program at low temperature. Corrected points of the program include:

(1) Increasing the number of preset temperatures from 10 to 20

(2) Automating the positioning for the origin of the sample holder

(3) Recording the temperature at the start and end of an XAS measurement in a log file

(4) Changing the width of the incident X-ray for each sample

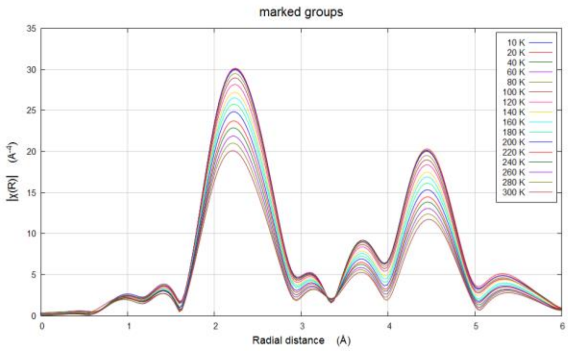

Fig. 5. Fourier transform of Mn K-edge EXAFS spectra for the Fe-Mn-C-Si martensitic steels by changing measurement temperature from 10 K to 300 K.

Figure 5 shows the Fourier transform of the Mn K-edge EXAFS spectra for the 0.1C-5Mn martensitic steel by changing the measurement temperature from 10 K to 300 K [3]. The intensity of each peak increases as the measurement temperature decreases due to the decrease of the Debye-Waller factor. The temperature of the start and end of an XAS measurement in a log file shows an error within 0.5 K for the preset temperature.

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References:

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[2] H. Ofuchi, et al., *SPring-8/SACLA Research Report* **5**, 207-211 (2017).

[3] T. Hanamura, et al., ISIJ International 53, 2218-2225 (2013).