Beamline practice at BL14B2 (XAFS)

H. Ofuchi, M. Takagaki, T. Honma (JASRI)

1. Introduction

The bending magnet beamline, BL14B2, is used for various applications of XAFS over a wide energy range from 3.8 to 72 keV. In the practical training course, we plan to show how to measure XAFS spectra, which covers alignment of X-ray optics and sample position. We will also demonstrate in-situ time-resolved quick scanning XAFS measurement of catalyst samples under reaction condition.

2. Plan of practice

9:30- Introduction of XAFS analyses

10:30- Preparation of tablet-form specimen.

11:30- Introduction of beamline and alignment of the x-ray optics.

12:30- ------- Lunch ----------

13:30- XAFS measurements of standard samples.

14:00- Introduction of preliminary process of XAFS spectra using software ATHENA.

14:30- Introduction of in-situ XAFS experiment

15:00- Measurement of in-situ XAFS spectra of catalysts during reduction process.
During the measurement, data treatment and preliminary analysis of XAFS spectra.

17:00 Close.

3. Alignment of x-ray optics and experimental stage

- Figure 1 shows schematic layout of BL14B2.
- Main operation in alignment of x-ray optics is follows.
  - Switch of diffraction plane of monochromator crystals between Si(111) and Si(311) to cover energy range for XAFS measurements.
  - Change of glancing angle of mirror to remove higher-harmonic x-rays.
- Above operation slightly changes beam height downstream of monochromator.
- Fine adjustment of slits and mirrors in the optics and experimental hutches is done by measuring x-ray intensity using 1st ionization chamber in the experimental hutch (Fig. 2).
- The height of experimental stage in the experimental hutch is adjusted to x-ray beam by scanning in vertical direction.
- Fixed beam condition is achieved by adjusting the rotation angle of 1st crystal of monochromator around beam axis to 2nd crystal.
- The control program for operation of x-ray optics realizes following x-ray beam.
  - Quite low contamination ($<10^{-4}$) of higher harmonics.
  - Fixed beam position during XAFS scans passing through the center of slits in the experimental hutch.
  - Focused beam in vertical direction (0.2-0.5 mm).

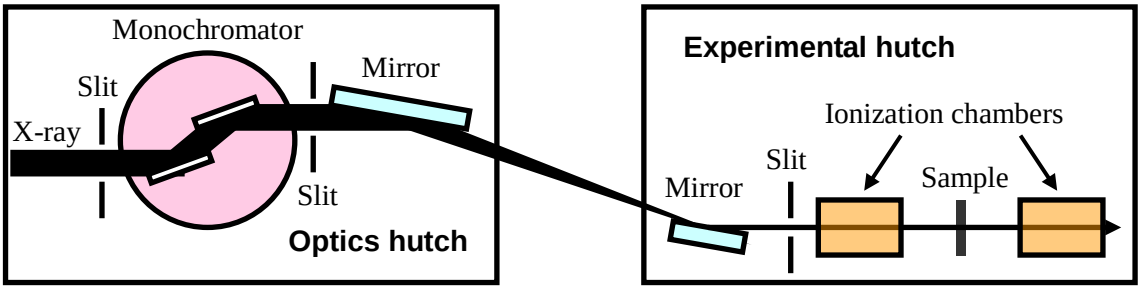

Fig.1 Schematic layout of BL14B2

4. Quick XAFS measurement in transmission mode

- Quick XAFS measurement is realized by data storage of x-ray detector output signals and Bragg angles of monochromator in each memory board during continuous scanning.
- The arrangement and control system for transmission mode Quick XAFS measurements are shown in Figs. 2 and 3.
- Setup of measurement system
  - Set the beam size incident on the sample by slit opening.
  - Set the gain of current amplifiers (Amp) for each ionization chamber.
- Operation of program of Quick XAFS measurement
  - Set the following parameters in the operation program
    - Energy range and step for a XAFS spectrum
    - Dwell time for a XAFS spectrum.

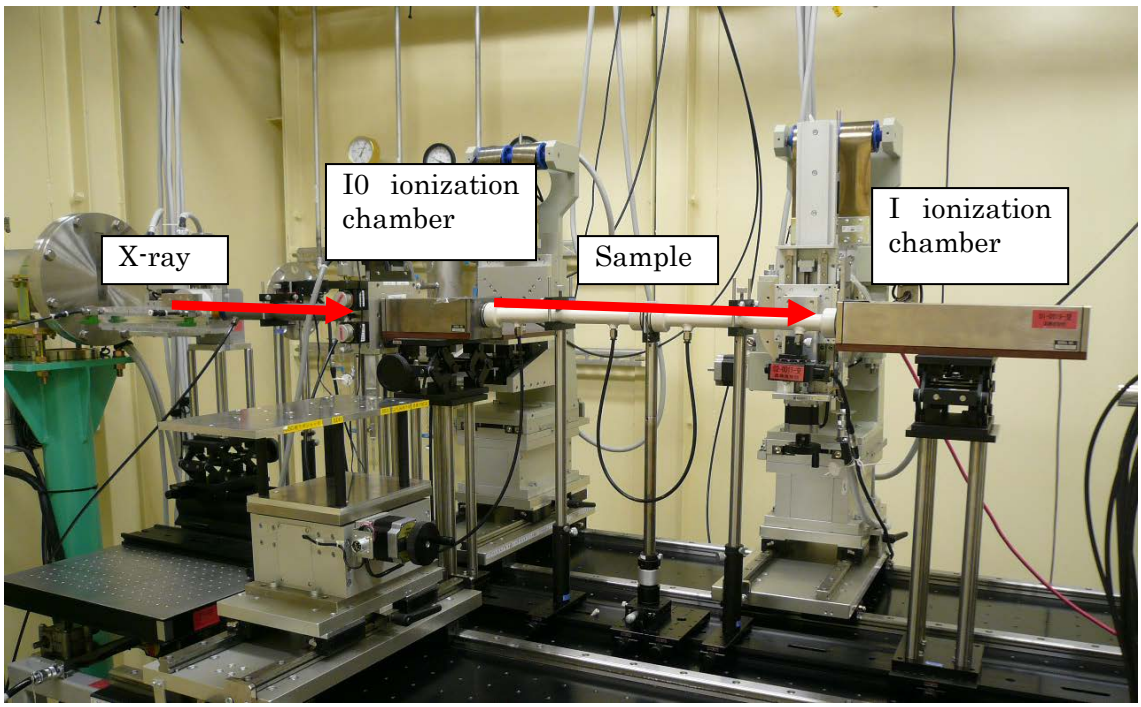

Fig. 2 Setup of transmission mode XAFS

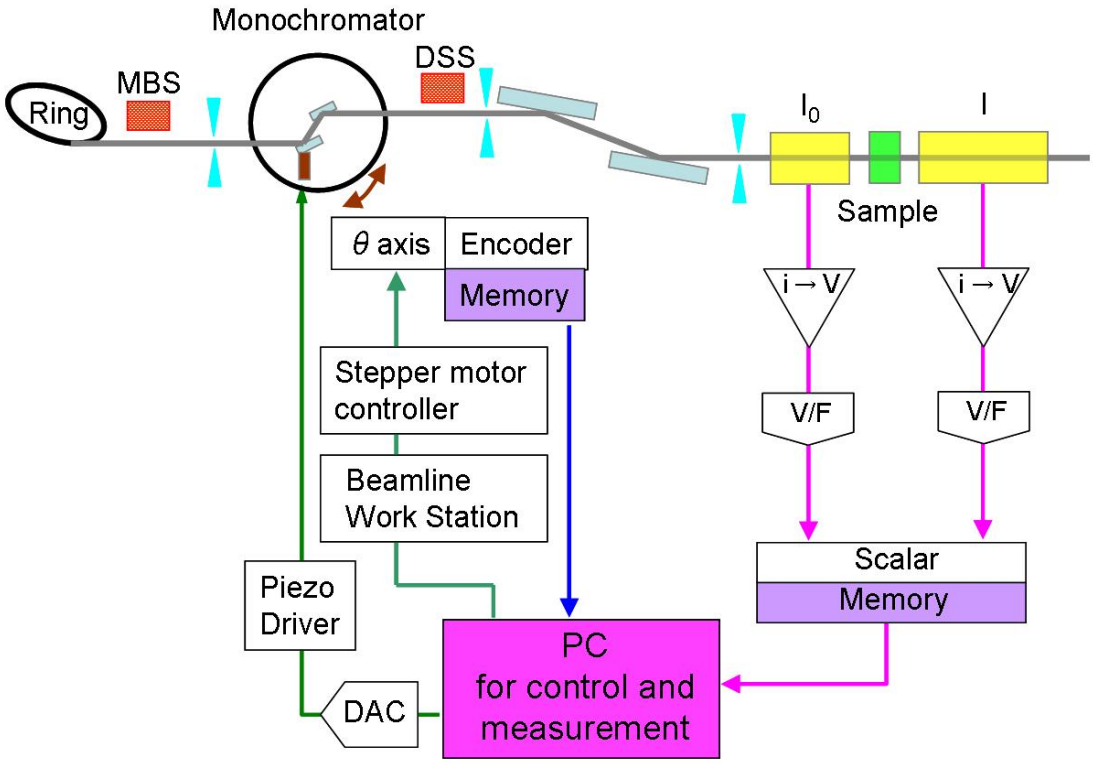

Fig. 3 Control system of QXAFS measurement system in transmission mode

5. Preliminary process of XAFS spectra using software ATHENA

- The row data of Quick XAFS spectra are processed to the ATHENA-readable data format.
- ATHENA processes the measured XAFS spectra, such as background removal and Fourier transforms.
  - ATHENA is one of most popular free software for processing XAFS data. http://cars9.uchicago.edu/~ravel/software/
  - Model fitting of XAFS data is done by another software ARTEMIS including interfaces to ATOMS and FEFF.

6. Measurement of in-situ XAFS spectra in transmission mode

6.1 Sample preparation

- The sample for XAFS measurement is form into thin disks of 7 mm diameter by press machine with very weak pressing force.
- Put adequate number of disk samples (around 20) into sample holder to achieve edge jump absorbance around 1.

Fig. 4 Mount of disk sample into inner holder of sample cell

6.2 Setup of in-situ XAFS equipments

- Experimental condition in this in-situ measurement
  - Sample: Pd/USY catalyst
  - Measurement energy: around Pd K-edge (24.3 keV)
  - Sample temperature: room temperature
  - Reaction gases: H_2, He and their mixture
- Set the sample cell including disk samples.
- Connect Q-mass to outlet of sample cell to monitor the concentration of each outlet gas.

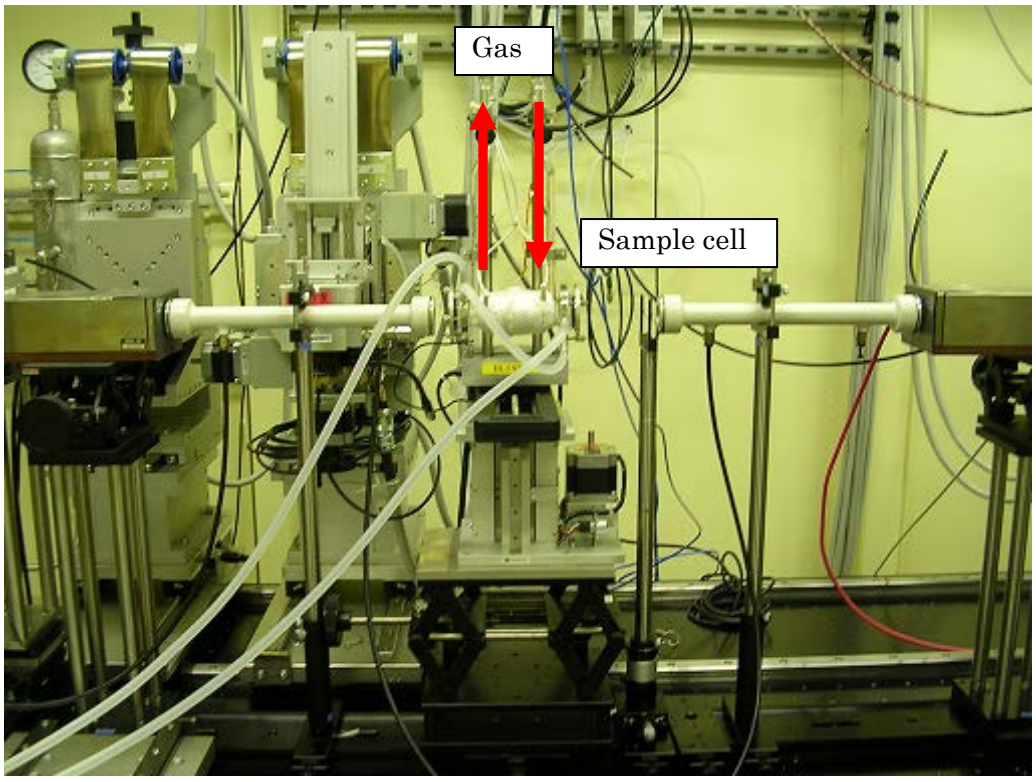

Fig. 5 Setup of in-situ XAFS equipments

6.3 In-stu XAFS measurement of catalyst during reduction process

- XAFS measurement of catalyst in initial state.
  - Flow 100%-He 100 ccm into sample cell.
  - Measure Quick XAFS spectra in transmission mode.
  - The measurement time of each XAFS spectra during this experiment is about 30 s.
- XAFS measurement of sample during reduction process.
  - Flow 8%-H_2/He 100 ccm into sample cell for 15-20 min.
  - Measure Quick XAFS spectra every 30 s during gas flow.
  - Check change of Fourier transform-XAFS spectra during reduction process using ATHENA.
  - Stop 8%-H_2/He flow.
  - Flow 100%-He for 15-20 min until no residual H_2 in sample cell (checking by Q-mass).
  - Measure Quick XAFS spectra.
  - Check the sample color.