BL14B1
QST Quantum Dynamics II

1. Introduction

BL14B1 is designed for various types of diffraction experiment and X-ray absorption fine structure (XAFS)-type spectroscopy measurements in the energy ranges of 5–90 keV for monochromatized beams and 5–150 keV for white beams. The main optics is the standard SPring-8 bending-magnet system with two mirrors and a fixed-exit double-crystal monochromator. These optical elements can be removed completely for an experiment involving white X-rays. This beamline has two experimental hutches. One is a white X-ray hutch dedicated to high-pressure and dispersive XAFS experiments with white X-rays. The other is a monochromatic X-ray hutch dedicated to XAFS, X-ray diffraction measurements, and X-ray irradiation experiments. BL14B1 can be a one-stop platform for the development of novel functional materials by the complementary use of white and monochromatized X-rays.

2. High-pressure and high-temperature experiments

High-pressure and high-temperature syntheses have been performed at the white X-ray hutch. *In situ* synchrotron radiation X-ray diffraction measurements can detect structural changes of a sample under high pressure and temperature. Consequently, the synthetic conditions of novel materials can be easily searched.

The $Y_{0.68}Mg_{0.32}Co_{3.00}$ alloy demonstrated a hydrogen storage capacity of 1.68 wt.% under a hydrogen pressure of 10 MPa. Notably, this capacity increased to approximately twice that value under a pressure of 10 GPa [1]. These findings suggest that, at 10 MPa, interstitial lattice sites capable of accommodating hydrogen atoms remain unoccupied. By promoting hydrogen incorporation into these sites—potentially through elemental substitution or other structural modifications—the hydrogen storage capacity may be further enhanced even under lower pressure conditions. This result represents a significant advancement toward the development of high-density hydrogen storage materials.

Other synthesis studies under high pressure have been carried out. Hydrogen-assisted Mg intercalation into TaS_2 has been reported, where the decomposition of MgH_2 was measured *in situ* [2]. The formation process of C_2Li has been investigated [3]. Microstructural changes occurring during high-temperature compression and subsequent isothermal holding processes—critical stages in the manufacturing of steel materials—were observed *in situ* using high-energy synchrotron X-ray diffraction with a two-dimensional detector, conducted within a high-temperature and high-pressure press apparatus [4].

3. Stress

Depending on the experiment's purpose, either monochromatic or white X-rays were used to evaluate material strength [5,6]. Suzuki *et al.* measured the residual stress present near the inner surface of the weld by the double-exposure method (DEM) with monochromatic X-rays. They also proposed a method involving a cross-correlation algorithm to accurately determine the diffraction angle from complex patterns arising from coarse grains, dendritic structures, and plastic zones. They also proposed a quantum beam hybrid method (QBHM) that uses circumferential residual stresses obtained by neutrons and DEM in a complementary manner. A residual stress map of the welded piping was obtained by QBHM, and a finite element analysis of the same butt-welded metal was performed. The results were then compared. A difference in results occurred because the measured residual stress map included the effect of the stress of each crystal grain as a result of elastic anisotropy, i.e., residual micro-stress.

4. XAFS

XAFS observation using an energy-dispersive optical system was carried out at the white X-ray hutch, as well as using a conventional optical system at the monochromatic X-ray hutch [7–10]. Various XAFS measurements, including those of high-speed chemical reactions and low-concentration additives, can be performed.

Several kinds of *in situ* observation equipment have been prepared in both the energy-dispersive and conventional optical systems. Remote control apparatuses such as gas flow controllers, switching valves, potentiostats, and injectors are always available. Some experiments using laser systems are also carried out. Time-resolved measurements are performed for reactions such as gas conversion, electrode, and ligand substitution reactions. In FY2024, the CO_2 hydrogenation reaction to methanol by Cu/ZnO catalysts was observed by time-resolved XAFS measurement at Cu and Zn K-edges under a 9 bar gas flow. The precise mechanism of the catalytic reaction under a high-pressure gas switching condition was studied by the simultaneous observation of Cu and Zn K-edges using dispersive optics.

In the conventional optical system, low-concentration XAFS measurements are performed using a 36-element solid-state detector. Local structural measurements of Cs-containing clay minerals at the Cs K-edge XAFS were carried out from the viewpoints of stable storage and volume reduction of radioactive wastes. A correlation between the flatness of the layered structure of weathered biotite clay and the Cs sorption site was detected by observing weathering-controlled samples and comparing them with clay minerals having different layered structures at 20 K. It was found that the existence of a layered structure that is partially broken is important for the stable sorption of cesium ions. We are continuing research to reveal the relationship between the structure of soil and the sorption state of cesium ions, which will lead to mobility evaluation and the selective collection of radioactive cesium ions.

5. X-ray irradiation on biological samples

Biological irradiation experiments using both white and monochromatic X-rays were started in FY2023. For the first time, animal experiments using mice were conducted at BL14B1 with ultrahigh dose-rate irradiation using white X-rays. Hair loss symptoms were observed when 20 Gy was irradiated in less than one second. Moreover, efforts are being made to irradiate biological samples with a microbeam of monochromatic X-rays. Irradiation with a 20 μm beam has been successfully achieved.

SAITOH Hiroyuki[*1], SHIRO Ayumi[*1], SHOBU Takahisa[*2], and MATSUMURA Daiju[*3]

[*1] Hydrogen Materials Research Group, Synchrotron Radiation Research Center, National Institutes for Quantum Science and Technology (QST)
[*2] Hierarchical Structure Research Group, Materials Sciences Research Center, Japan Atomic Energy Agency
[*3] Advanced Energy Materials Research Group, Materials Sciences Research Center, Japan Atomic Energy Agency

References:
[1] Sato, T. *et al.* (2025). *J. Phys. Chem. C* **129**, 2865.
[2] Fujioka, M. *et al.* (2024). *J. Am. Chem. Soc.* **146**, 34324.
[3] Mukai, K. *et al.* (2024). *Sci. Rep.* **14**, 26304.
[4] Yonemura, M. *et al.* (2025). *Metall. Mater. Trans. A* **56**, 1193.
[5] Hojo, T. *et al.* (2024). *ISIJ Int.* **65**, 284.
[6] Suzuki K. *et al.* (2025). *Quantum Beam Sci.* **9**(2), 15.
[7] Kobayshi, T. *et al.* (2024). *Dalton Trans.* **53**, 18616.
[8] Krajewska, A. *et al.* (2024). *Sci. Adv.* **10**, eadn3880.
[9] Jung, H. *et al.* (2024). *ACS Energy Lett.* **9**, 2162.
[10] Lei, Y.-J. *et al.* (2024). *Nat. Commun.* **15**, 3325.