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 refers to 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 developments of novel functional materials by the complemental 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. Additionally, the system can investigate the reaction mechanisms [1–7].

Currently, research focuses on synthesizing novel hydrides. In FY2021, a novel Al-Fe alloy hydride was investigated [8]. $Al_{13}Fe_4$ alloy was hydrogenated at 9 GPa and 750°C. Its hydrogenation process was monitored using an in situ synchrotron radiation X-ray diffraction system at BL14B1. In situ measurement revealed that the $Al_{13}Fe_4$ alloy was hydrogenated above 700°C at 9 GPa in fluid hydrogen to form the novel hydride Al_3FeH_4. The formed hydride can be recovered under ambient conditions. The recovered Al_3FeH_4 evolved hydrogen above 150°C at ambient pressure, and the hydrogen concentration was calculated to be 2.9 wt% on the basis of the weight loss during the hydrogen evolution. It is expected that the hydride can be used as a low-cost hydrogen storage material if we can reduce the hydrogenation pressure.

3. Stress

Previous studies have shown that the double-exposure method (DEM) is very effective for the stress measurement of materials with coarse grains. In FY2021, we applied the DEM using high-energy synchrotron radiation monochromatic X-rays, which was not used in the past, and conducted stress measurements of a shrink-fitted ring. The grain size of this sample was 43 μm, and diffraction rings were spotty as diffraction from the sample was observed with a 2D CdTe pixel detector. By applying the DEM and obtaining a good diffraction curve, we were able to calculate the strain even at diffraction spots. From these results, it was clarified that the DEM using high-energy monochromatic X-rays is also effective for coarse grains [9].

Resistance spot welding is commonly used as a joining method in automotive manufacturing.

However, there are issues such as weld defects and insufficient joint strength in dissimilar material joints. In FY2021, we focused on zinc embrittlement cracking (LMEC), which occurs on the tangential surface of welds, and clarified its occurrence by imaging techniques using high-energy white X-rays at BL14B1. Image reconstruction showed that cracking was suppressed as the pressurization holding time increased.

4. XAFS

XAFS observation using an energy-dispersive optical system was operated at the white X-ray hutch as well as a conventional optical system at the monochromatic X-ray hutch [10–13]. Various XAFS measurements such as those involving high-speed chemical reactions and low-concentration additives can be performed.

Several in situ observation devices have been prepared in the energy-dispersive optics hutch. Remote control systems such as gas flow controllers, switching valves, potentiostats, and injectors are always available. Some experiments using laser systems are also operated. Time-resolved measurements are performed for gas conversion reactions, electrode reactions, ligand substitution reactions, and so forth. In FY2021, the deuteration reactions of Rh and Pd metal nanoparticles were observed by time-resolved XAFS measurement at a rate of about 50 Hz, and the reaction mechanism of deuterium molecules was clarified from the distortion of nanoparticles.

In the conventional optical system, low-concentration XAFS measurements are performed using a 36-element solid-state detector. For example, local structure measurements of Cs-, including clay minerals, at Cs K-edge XAFS were carried out from the viewpoints of stable storage and volume reduction of radioactive wastes. A correlation between the layered structure of biotite clay and the Cs sorption site was detected by the observation of the weathering-controlled samples. We are continuing research to reveal the relationship between the structure of soil and the sorption state of cesium ions, thereby leading to the mobility evaluation and selective collection of radioactive cesium ions.

5. X-ray irradiation effects on a tumor

The X-ray irradiation effects on a tumor were investigated for Auger therapy. Tumor spheroids containing I-loaded nanoparticles were irradiated with synchrotron radiation monochromatic X-rays. The destruction mechanisms of tumor spheroids containing nanoparticles were investigated. To clarify the destruction mechanisms, we evaluated the absorbed dose in our system by calculating the dose from the number of X-ray photons and measuring it using Fricke dosimeters. Both the obtained values agreed well, within the experimental errors.

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