Poster Presentation

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Time-resolved Powder Diffraction System with Gas Control at BL02B2/SPring-8

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Obtaining fundamental structural information on gas molecules adsorbed in nanopores of porous materials plays important roles for the understanding of the adsorption/desorption processes and the rational synthetic design of functional materials for gas storage, gas separation, catalytic reaction, etc. Rietveld and maximum entropy methods for synchrotron powder diffraction data in gas adsorption condition revealed the static crystal structured of metal-organic frameworks with the adsorption of gas molecules [1,2]. The diffraction data was collected using imaging plate detector installed on a large Debye-Scherrer camera at third-generation synchrotron radiation facility, SPring-8. However, high-resolution diffraction data collection within a second is necessary to obtain in-situ structural information in adsorption/desorption processes. To establish the trend of the structural information under controlled gas atmosphere, we developed time-resolved powder diffraction measurement system under remote-controlled gas pressures. This system consists of six MYTHEN detectors (Dectris Ltd., Baden, Switzerland), remote-gas handling system, capillary gas cell, and wide-range temperature control systems at beamline BL02B2 of SPring-8 [3]. MYTHEN is a one-dimensional X-ray detector operating in single-photon-counting mode, and each module consists of 1280 50-μm-pitch silicon micro-strips that serves as X-ray sensors. The maximum frame rate is approximately 17 fps. The gas or evacuated atmosphere is controlled by the developed handling system. This gas handling system consists of a digital mass flow controller, air valves, Baratron vacuum gages, and a turbo-molecular pump. The pressure and gas flow rate in the capillary sample can be automatically controlled in the 0.1 Pa – 130 kPa pressure range by the developed remote control software. Using developed apparatus, we performed time-resolved powder diffraction measurements for metal-organic frameworks under O2 gas pressures. The data acquisition time was 0.5 seconds, and the data are recorded simultaneously at 2θ values ranging from 2° to 38° in one-shot measurements without some gaps between the detector modules. The sample was cooled down to 95 K by N2 gas stream device, and the adsorbed gas was introduced into capillary sample gas cell via remote-handling system. The pressure range was controlled from 0.1 Pa to 80 kPa during the measurement. On these experimental conditions, novel intermediate adsorption phases were successfully observed, and we revealed that the formation of the phase is dependent on the gas pressure and adsorption/desorption speed.

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