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Observation for materials constructed from light elements by phase-contrast X-ray imaging under low and high temperature conditions

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1 Introduction

Application ranges of organic compounds composed of light element, such as carbon, nitrogen and oxygen, and their operating environments are enormous. Phase-contrast X-ray imaging (PCXI) technique with synchrotron X-ray radiation is a unique technique to visualize light element materials and low-density materials with high resolution [1], whereas a conventional absorption-contrast X-ray imaging technique is not suitable to visualize light element materials. X-ray interferometric imaging (XII) and diffraction enhanced imaging (DEI) are the two major techniques of PCXI for two- and three-dimensional observations of samples with several cm in size. The density resolution of XII is higher than that of DEI. XII is suitable for detecting gradual phase-shifts and the absolute density of materials, but the observations are limited by a sample-outlining effect that occurs from the steep phase-shifts caused by density difference between the sample and air. On the other hand, DEI offers a wide density dynamic range, which enables observation of samples containing regions with large density gradients [2].

So far, application of these imaging techniques have been limited for observations under room temperature condition because of the difficulty to control temperature of samples without interrupting measurements of X-ray phase-shift. We have developed a cryo-chamber for XII measurements at temperatures below room temperature down to about 200 K. This allows observations of gas hydrate such as air hydrate within ice core drilled from the Antarctica [3]. On the other hand, developments of observation methods for the assessment of materials changes over time under various temperature conditions have been required for various organic compounds.

In this paper, a temperature-controlled chamber for PCXI measurements by means of XII and DEI is reported. The temperature of the chamber was kept at setting values from 190 K to 370 K.

2 Design

We designed a temperature-controlled chamber for PCXI experiments with synchrotron radiation (See Fig. 1). The chamber was made of aluminium with a size of W210 × L165 × H230 mm^3. X-ray window was put at each end of the sample chamber, and the size of these windows was adjusted to 60 × 30 mm^2 for XII measurements using a two-crystal X-ray interferometer so that the object X-ray beam and the reference X-ray beam could go through them.

Inside the temperature-controlled chamber, a sample container made by copper and having two X-ray windows of 25 × 35 mm^2 in size at each end was mounted (See Fig. 2). For XII a measurement, the sample container was filled with unfrozen liquid over the temperature range used, i.e. Methyl acetate for gas hydrate observations. The liquid around the sample prevented undesirable outline contrasts from the outer surface of the sample, allowing internal observation. All of X-ray window was made of aluminium foil with a thickness of 0.5 mm because high-energy X-ray of 35 keV was used herein.

Temperature was monitored by a thermocouple attached to the center of the sample container. The temperature was controlled by coordinating temperature and flow rate of blowing dry N_2 gas into the temperature-controlled chamber. In addition, two sheet heaters attached to the both ends of sample container and controlled by PID controller were used to control the

Fig. 1: Custom temperature-controlled chamber installed in the diffraction enhanced imaging (DEI) system.

temperature. Consequently, the temperature of the chamber was kept at setting values from 190 K to 370 K for more than 24 hours with temperature deviation of about 1 K.

The temperature-controlled chamber was insulated by a vacuum layer, and the temperature outside was maintained at room temperature. The X-ray windows were composed of a two-layer structure, and the layer was also vacuumed for insulation. The insulation prevented condensation from occurring on the windows due to the difference between the room temperature and the low temperature in the chamber. Thus, the chamber did not disturb the measurement of the X-ray phase shift using an asymmetric crystal and an analyzer crystal.

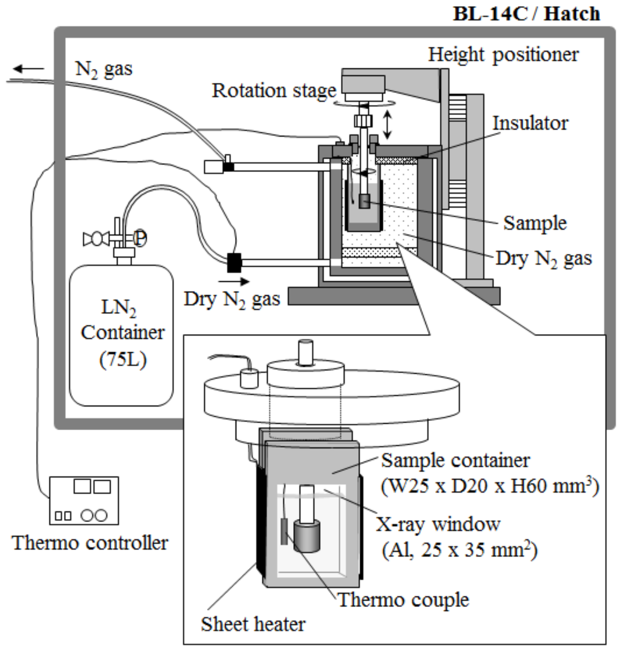

Fig.2 Experimental setup for temperature-controlled phase-contrast X-ray imaging measurements.

3 Application

We have developed and modified the temperature-controlled chamber for PCXI measurements performed at beam line (BL-14C) of the Photon Factory in Tsukuba, Japan. So far, we reported applications of the system to gas hydrates [3-7] and carbon paper that can be used for fuel cells [8-9]. Now, it is possible to control various temperatures for more than 24 hours, which may be useful to observe time dependence of samples under isothermal conditions. Also, the temperature deviation of about 1 K of the established temperature-controlled system may be acceptable for *in-situ* observations for temperature-dependent process of materials. This imaging system reported herein may open up new applications of phase-contrast X-ray imaging techniques.

References

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