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Simultaneous measurement of SAXS and absorption factor for supercritical fluid by use of Si PIN photodiode installed in direct beam stopper

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Introduction

Determination of absorption factor, μl, is usually difficult in diffraction experiments in high temperature and high pressure conditions, where μ and l are linear absorption coefficient and path length, respectively. Small-angle X-ray scattering (SAXS) pattern and the magnitude of the scattering intensity sensitively depend on correction of background intensity and absorption, both of which are made by use of absorption factor. Therefore, the accurate determination of the factor is of particular importance for SAXS experiments. Our method for the determination of the factor, which is simply constructed by a beam stopper with a pinhole and a one dimensional detector, is described previously.[1] However, the method is not enough in stability and accuracy. Here we report on the apparatus for measurement of the factor by use of photodiode installed in direct beam stopper. The apparatus enables us to measure intensities of SAXS and transmittance of the sample simultaneously, without increase of parasitic scatterings.

Experimental

At BL-15A, simultaneous measurements of SAXS and absorption factors of a sample fluid were carried out for supercritical water along an isotherm at 674.3 K and pressures between 23.3 and 31.4 MPa. The corresponding density values are from 0.136 to 0.394 g/cm^3. Scattered X-rays were detected by position sensitive proportional counter (PSPC). The transmittance of direct beam was monitored by Si PIN photodiode (Hamamatsu Photonics, S5106) connected to a picoammeter (Keithley Instrument, 6485). Because the device was installed in direct beam stopper in vacuum chamber, there was not increase of background due to path of air and gas for ionization chamber. Restraint of background is essential for precise SAXS measurement in small s-region (s: $4\pi\sin\theta / \lambda$).

Result and discussion

Figure 1 shows the absorption factor at various pressures during the exposure time. A supercritical fluid is a suitable standard for confirmation of validity of the measurement, because the fluid varies its density widely with modest changes of temperature and/or pressure form gas-like to liquid-like values. As shown in Fig. 1, in wide density range, the measurements were performed stably. Figure 2 (a) and (b) show SAXS intensities, for which correction of subtraction and absorption are made by the measured absorption factors, and the Ornstein-Zernike plots, $1/I(s)$ vs. s^2, at typical states, respectively. In small-s region of the plot shown in Fig. 2(b), deviation from linear relation was observed, especially at 24.7 MPa. This indicates that the estimated values of the absorption factor might be smaller than accurate values. In fact, the measured absorption factors show agreement with those determined in laboratory by X-ray absorption experiment within 10 % error. It is concluded that the apparatus provides indication of absorption factor by *in-situ* measurement, and further improvement is necessary for accurate determination.

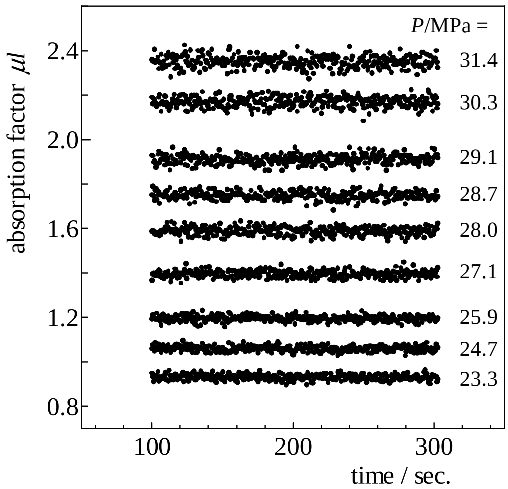

Fig. 1 Absorption factor μl simultaneously measured by Si PIN photodiode installed in direct beam stopper.

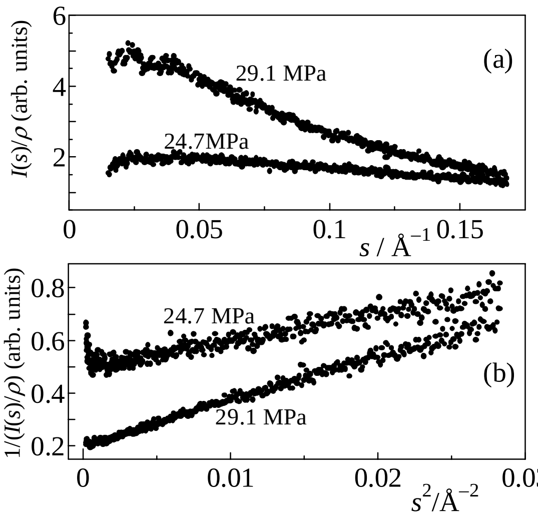

Fig. 2 (a) SAXS intensities and (b) Ornstein-Zernike plots for supercritical water at 674.3 K and 24.7, 29.1 MPa.

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References

[1] K. Nishikawa *et al*., J. Phys. Chem. B101, 1413 (1997).

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