Fabrication of a Compact Transfer Vessel System for Anaerobic Samples at BL-2 and BL-10

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1. Introduction

A compact transfer vessel system for anaerobic samples, for example, a lithium ion battery was fabricated for the two X-ray absorption fine structure (XAFS) beamlines; ultra-soft XAS beamline (BL-2) and soft x-ray XAFS beamllne (BL-10) in the SR center, Ritsumeikan University [1]. Both BL-2 and BL-10 can use the same transfer vessel by using a same fundamental set up of the system. In this paper, an outline of the system and the result of an evaluation measurement which was carried out at BL-10 are reported.

2. Outline of the Transfer Vessel System

The transfer vessel system mainly consists of a load-lock chamber (with a high vacuum pumping system), two transfer rods, one for sample transfer and another for sample lifting, and a transfer vessel (see Fig. 1). The transfer vessel consists of an ICF70 UHV gate valve, a one-side-sealed pipe, a cover with leak valve with a NW40 quick coupling. Inside the one-sided-sealed pipe, a sample rack for 8 sample holders is loaded (see Fig. 2).

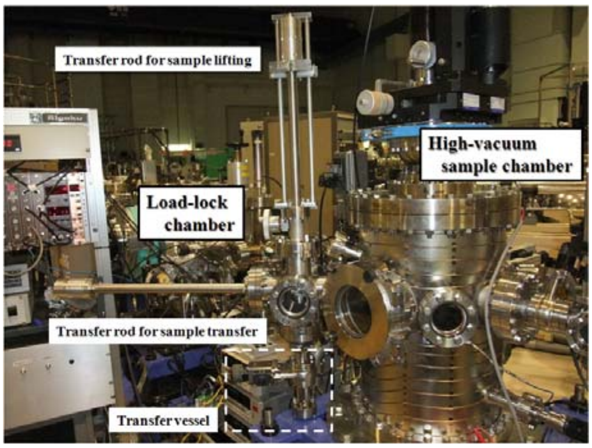

Fig. 1. Sample chamber and load-lock chamber for developed transfer vessel system at BL-10.

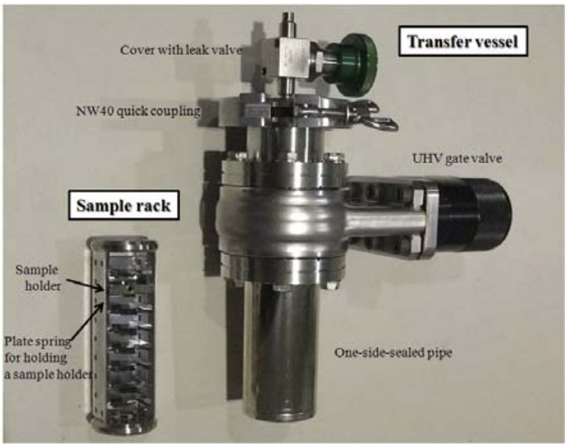

Fig. 2. Transfer vessel and sample lack.

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In this system, it is assumed that the sample preparation and sample loading in the transfer vessel are done under Ar gas atmosphere in a glove box. After sample loading, the UHV gate valve should be closed tightly, and additional valve in the other side prevents from air or water invasion. For further protection, sealing the whole transfer vessel in an aluminum laminated foil box might be preferable especially when it takes long time to bring it to the center.

The sample plates are shown in Figs. 3 and 4. A sample must be put on the area within 20 mm × 20 mm of the sample plate. It is preferable to make arrangements with the person in charge of the beamlines in advance, because each beamline has its own recommended sample size, measuring method, *etc*. For example, in the case of BL-10, the sample size of about 15 mm (vertical) × 5 mm (horizontal) is needed, because the X-ray size at the sample position is about 5 mm (vertical) × 2 mm (horizontal), and a sample is rotated horizontally to use an X-ray detector.

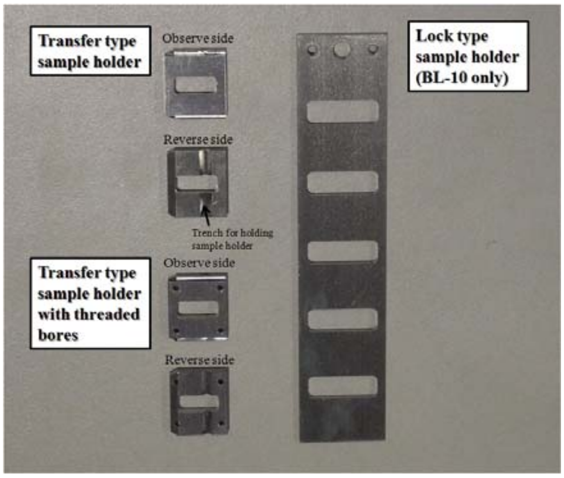

Fig. 3. Photograph of sample holders.

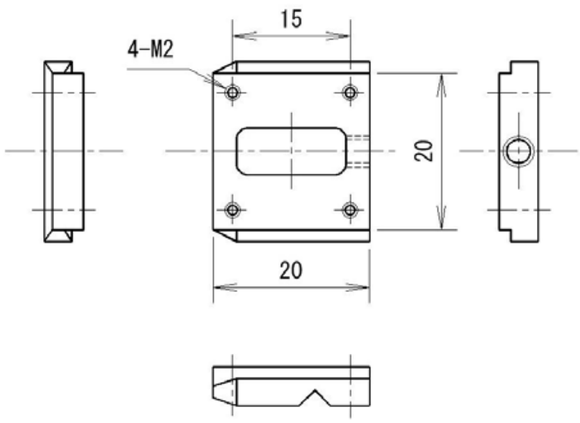

Fig. 4. Drawing of a sample holder (with threaded bores).

3. Performance test of the transfer vessel

The performance of the transfer vessel system was examined by using $MgCl_2$ which is known as a highly deliquescent material. It changes from a nonhydrate to a hydrate quite easily by exposing to air. Fine $MgCl_2$ powders were put on the sample plate using a carbon tape and evacuated in the transfer vessel to change from hydrated $MgCl_2$ by air exposure to nonhydrate completely [2]. After evacuating for a few hours, $MgCl_2$ was purged by Ar gas and UHV gate valve of the transfer rod was closed. The $MgCl_2$ sample was kept in the transfer vessel for 2 days ('2 days kept in vessel'). To compare this $MgCl_2$ sample, a $MgCl_2$ sample just after evacuation ('no air exposure'), a $MgCl_2$ sample exposed to air for 10 seconds ('air exposure for 10 sec.'), and a $MgCl_2$ sample exposed to air for 30 minutes ('air exposure for 30 min.') were prepared.

Mg K-edge X-ray absorption near edge structure (K-XANES) measurement was carried out under atmospheric pressure in helium gas at BL-10 by partial fluorescence yield (PFY) using a silicon drift detector. Observed Mg K-XANES spectra were shown in Fig. 5. A self-absorption effect of PFY is not corrected for these spectra. A spectral shape of 'No air exposure' differs from that of 'air exposure for 10 sec' and 'air exposure for 30 min' obviously. This shows that a $MgCl_2$ powder is easily changed from a nonhydrate to a hydrate by exposing to air for a little time. '2 days kept in vessel' spectrum is very similar to 'no air exposure'. This result shows the $MgCl_2$ sample kept in the transfer vessel did not change at all in 2 days.

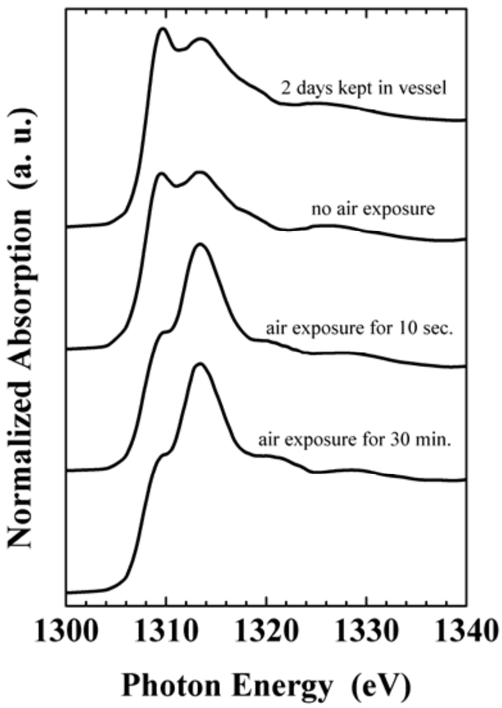

Fig. 5. Observed Mg K-XANES spectra of $MgCl_2$ in various conditions.

In this study, the $MgCl_2$ sample was kept the condition without the cover and an aluminum laminated foil. However, in the case of a practical sample, we think that the cover and an aluminum laminated foil should be used in order to minimize a risk, such as an air exposure, a shock during transportation.

4. Summary

We developed a compact transfer vessel system for anaerobic samples. This can be used as a common system at BL-2 and BL-10 of the SR center, Ritsumeikan University. The transfer vessel was verified by using $MgCl_2$ and the sample in the transfer vessel was kept the condition for 2 days. We expect this transfer vessel is used by many users and connects with many results of research.

References

[1] Koji Nakanishi, Shinya Yagi, Toshiaki Ohta, *IEEJ Trans. EIS*, in Press.

[2] Koji Nakanishi, Shinya Yagi, Toshiaki Ohta, *AIP Conf. Proc.* **1234** (2010) 923-926.