CROSS TOKAI **Experimental Report** J-PARC	承認日 Date of Approval 2014/08/21 承認者 Approver Kaoru Shibata 提出日 Date of report 2014/8/21
実験課題番号 Project No. 2013I0102 実験課題名 Title of experiment Development of sample environmental equipment including high pressure and gas atmosphere introduction 実験責任者名 Name of principal investigator Takeshi Yamada 所属 Affiliation CROSS-Tokai	装置責任者 Name of Instrument scientist Kaoru Shibata 装置名 Name of Instrument/（BL No.） DNA / BL02 利用期間 Dates of experiments 2013/04/09 09:00 ~ 2013/04/12 07:00 2014/03/27 21:00 ~ 2014/03/30 21:00 2014/03/21 09:00 ~ 2014/03/22 09:00

1．研究成果概要（試料の名称、組成、物理的・化学的性状を明記するとともに、実験方法、利用の結果得られた主なデータ、考察、結論、図表等を記述してください。

Outline of experimental results (experimental method and results should be reported including sample information such as composition, physical and/or chemical characteristics.

The aim of this CROSS Development Project is to perform the development and to do test experiments for the following two type sample environment equipment as taking advantage of the characteristics of high energy resolution spectrometer DNA (BL02).

(A). An in situ water vapor and gas atmosphere introduction equipment

(B). A high-pressure generation equipment (P< 2000 atm)

In 2013 period, the water vapor introduction experiments with the gas introduction equipment were performed as described below. The high-pressure generation equipment was prepared in the 2013 year CROSS budget. However, time of the pre-test is not enough thus, the beam experiment was not performed in 2013. This will be performed in 2014.

The water vapor introduction experiments on Nafion® membrane were performed by using the gas atmosphere introduction equipment mounted on the BL02 top-loading cryostat and using BL02 spectrometer with 13 μeV energy resolution. The schematic image of the gas atmosphere introduction system is shown in Fig. 1. The vapor pressure was controlled by the auto control pressure regulated valve (valve P on Fig. 1).

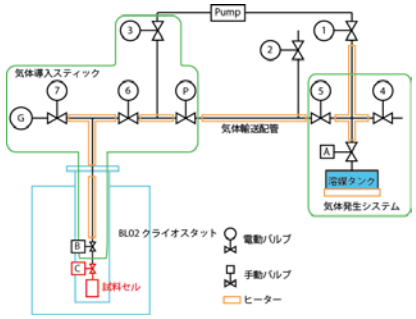

Fig. 1. Schematic image of the gas atmosphere introduction system.

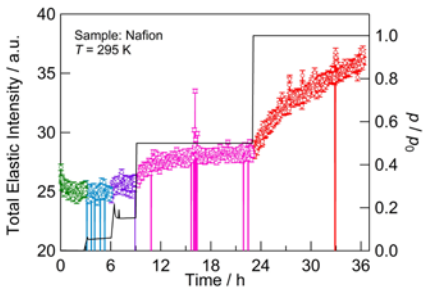

Fig. 2. Plots of elastic intensity and setting relative pressure of the water vapor.

1．研究成果概要（つづき） Outline of experimental results (continued).

Fig.2 shows elastic intensity and setting relative pressure of the water vapor to the time. The elastic intensity increased with increasing the relative pressure. This result indicated that the water vapor introduction succeeded. The recorded date at p / p_0 = 1.0 were also sliced each 1 or 2h in order to evaluate the time evolution of the QENS patters.

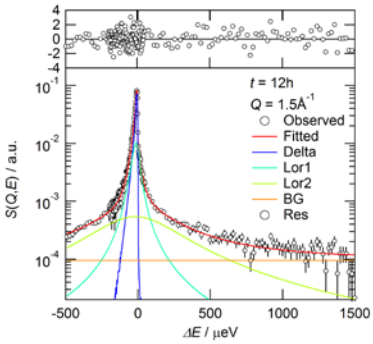

Fig. 3. QENS profile at p / p_0 = 1.0 after 12 h.

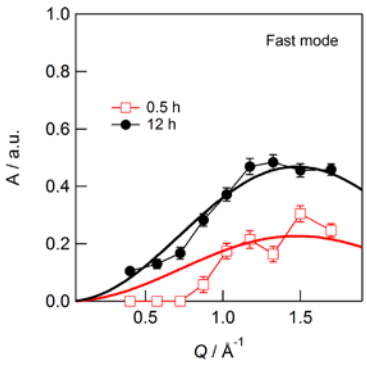

Fig. 4. Plot of intensities of the Lorentz function for the fast mode on 0.5 and 12 h.

Fig. 3 shows QENS profile at p / p_0 = 1.0 after 12 h. The time width was 2h. The enough statistics to analyze the data was obtained. The profile was well fitted by sum of a delta function, two Lorentz functions corresponding slow and fast modes, respectively and constant background convoluted with the resolution function. The Q dependence of the half width at half maximum of the both modes was almost constant in the measured Q range (not shown here) and the observed modes might be due to the localized mode. Fig. 4 shows Q dependence of the intensity of the Lorentz function for the fast mode. The data were fitted by the first-order spherical Bessel function in order to evaluate the intensity. The obtained pre-factor of the fitted function was shown in Fig. 5. The intensity of the fast-mode corresponding to the motion of the free water was saturated after 6 h otherwise, the intensity of the slow mode corresponding to the confined water continued to increase. This result indicated that the structure of the hydrophilic domain keep changing during the adsorption in measured time range.

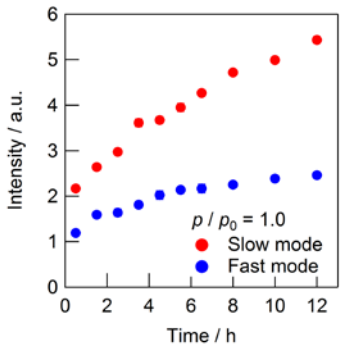

Fig. 5. Time evolution of the pre-factor of the spherical Bessel function for the slow and fast modes.

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