TOKAI CROSS Experimental Report J-PARC	提出日 Date of Report 2012/10/22
課題番号 Project No. 2012A0111 実験課題名 Title of experiment Characterization of water-saturated compacted montmorillonites by small-angle neutron scattering 実験責任者名 Name of principal investigator Hiroaki Takahashi 所属 Affiliation Japan Atomic Energy Agency	装置責任者 Name of responsible person Jun-ichi Suzuki 装置名 Name of Instrument/（BL No.） TAIKAN (BL No. 15) 実施日 Date of Experiment 2012/6/3 - 2012/6/4

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Used as samples is montmorillonite, Kunipia-F (the montmorillonite content is >99 wt.%), which is commercially available from Kunimine Industries, Japan. The chemical formula of the montmorillonite is $Na_{0.42}Ca_{0.068}K_{0.008}(Al_{1.56}Mg_{0.31}Fe(III)_{0.09}Fe(II)_{0.01})(OH)_2(Si_{3.91}Al_{0.09})O_{10}$.

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Experimental method

The experiments were performed with 20-mm-diameter and 2-mm-thickness samples of deuterated water-saturated compacted montmorillonites with dry density of 0.08, 0.4, 0.6, 0.8, 1.0, 1.2 and 1.4 Mg m^{-3}. The saturation of montmorillonite was carried out in a vacuum chamber to accelerate the saturation and to remove air bubbles for more than 1 month.

Small Angle Neutron Scattering.

The SANS experiments were performed with TAIKAN/BL15 diffractometer. The used beam band was from 0.6 to 7.3 Å, which corresponds to the Q range from 0.02 to 0.6 Å with the then data acquisition. Each sample was exposed incident neutron beam for two hours.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

Results

As Figure 1 shows, intensity decreases continuously with increasing Q for all deuterated water-saturated compacted montmorillonites. As a general observation, intensity does not reach a plateau value at low Q and shows Q^{-2} slope. This experimental evidence may be ascribed to the form factor of the clay thin sheets with the diameters larger than $2\pi/q_{min} \approx 300$ Å.

The swelling of montmorillonite has been designated as crystalline swelling for which *d*(001) is less than 19 Å and osmotic swelling due to the development of diffuse double layers within the interlayer regions leading to *d*(001) values which may be considerably greater than 19 Å. In the case of dry density of 0.4 Mg m^{-3}, the peak corresponding to the osmotic interlayer swelling at Q = 0.087 $Å^{-1}$, which is 72 Å of *d*(001) value was observed. Additionally, the *d*(001) values decreased with increasing dry density of compacted montmorillonite to 40 Å for 1.0 or 1.2 Mg m^{-3}. The peak corresponding to the osmotic interlayer swelling was not observed at dry density of 1.4 Mg m^{-3}. This result and our XRD data indicate that only crystalline interlayer swelling is existed at dry density of 1.4 Mg m^{-3}. The peak corresponding to the osmotic interlayer swelling was also not observed at dry density of 0.08 Mg m^{-3}. This result indicates that exfoliation into single sheets became prevalent while the swollen component declined at dry density of 0.08 Mg m^{-3}.

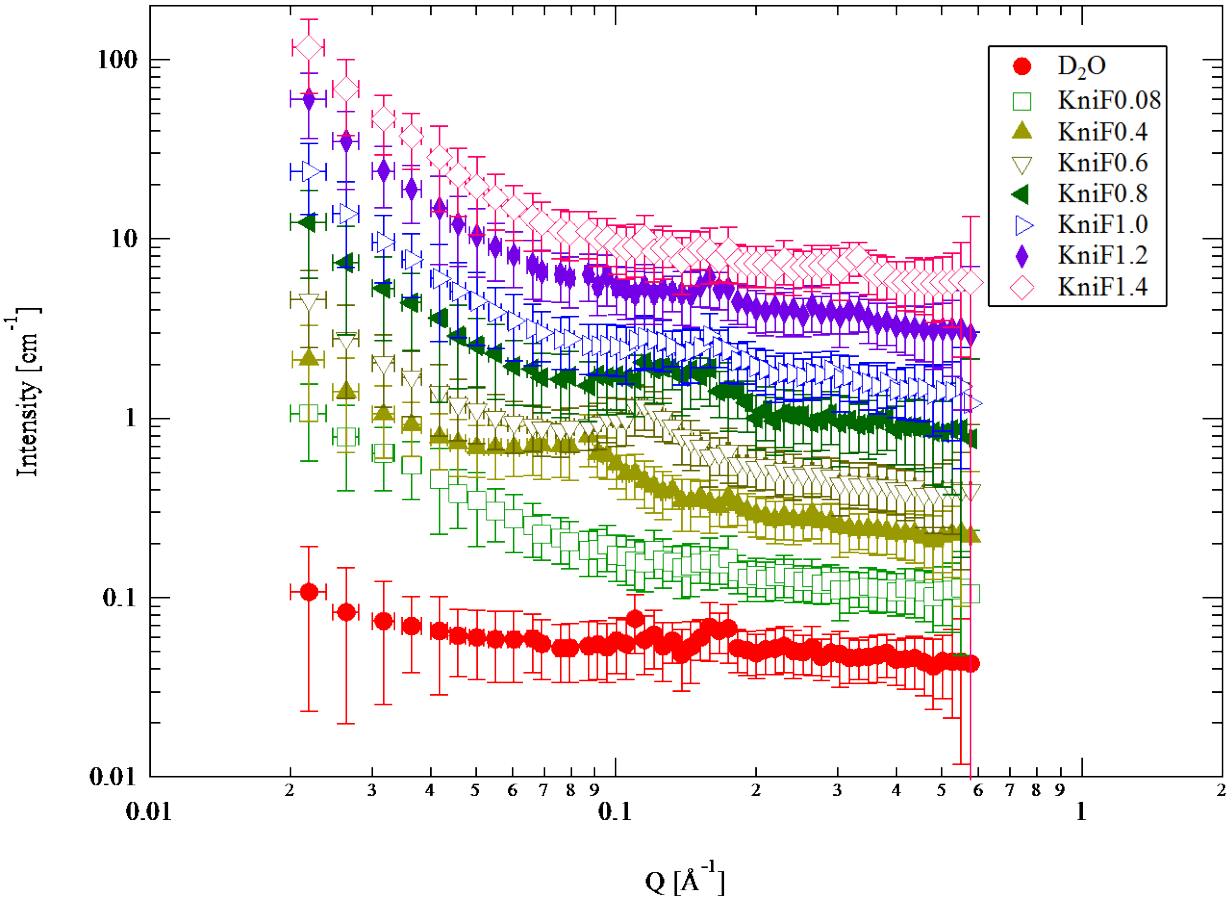

Figure 1. Scattering function of deuterated water-saturated compacted montmorillonite with dry density of 0.08, 0.4, 0.6, 0.8, 1.0, 1.2 and 1.4 Mg m^{-3}. Scattering functions have been magnified in intensity by a factor of two, except in the case of KuniF0.8 by a factor of four to make these patterns clearly visible.