MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 2012A0124 実験課題名 Title of experiment Structural analysis of high-capacity negative electrode materials for Li-ion battery 実験責任者名 Name of principal investigator Yohei ONODERA 所属 Affiliation Kyoto University	装置責任者 Name of responsible person Toshiya Otomo 装置名 Name of Instrument/（BL No.） NOVA (BL21) 実施日 Date of Experiment 2012/10/26-28

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.

Amorphous silicon, a-Si

Amorphous Li-Si alloy, xLi-(1-x)Si (x=0.1, 0.3, 0.5)

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.

In this proposal, we carried out neutron total scattering experiments in order to study the local structure of Li-Si amorphous materials, which is known as promising high-capacity negative electrode materials for Li-ion batteries.

The xLi-(1-x)Si amorphous samples were synthesized by the mechanical alloying (MA) method from Li metal foil and amorphous silicon (a-Si) powder prepared by vapor deposition technique. The starting materials were put into a zirconia pot with 20 zirconia balls under high purity argon gas atmosphere, and then alloyed mechanically at 150 rpm for 10 h using a planetary ball mill apparatus (Fritsch, P-5).

The neutron total scattering experiments were carried out with the High Intensity Total Diffractometer (NOVA), installed at BL21 of MLF in J-PARC. The powder samples by MA xLi-(1-x)Si (x=0.1, 0.3, 0.5), and amorphous Si were put into cylindrical vanadium-nickel cells (6 mm in diameter) under argon gas atmosphere. The neutron total scattering data of the samples were measured for 6 h at room temperature with 200 kW of MLF beam power. Structure factors, $S(Q)$, were obtained from the scattering intensity after correction of the background, attenuation, and multiple scattering and normalization with scattering from a vanadium rod.

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

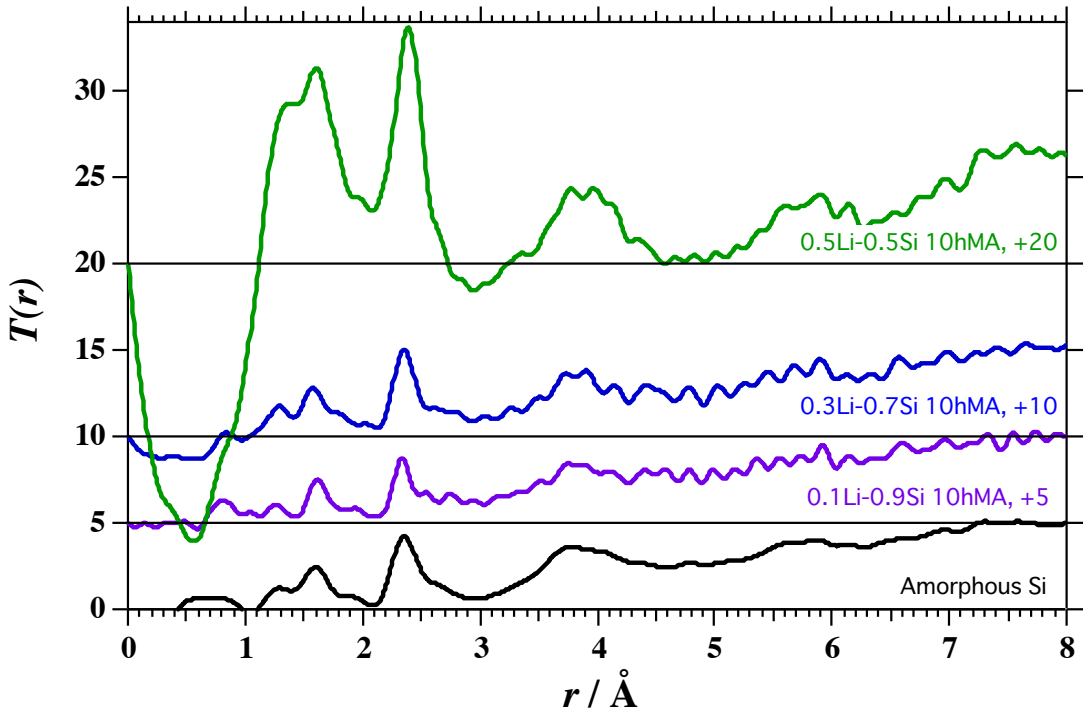

Fig.1 xLi−(1−x)Si アモルファス試料の全相関関数 $T(r)$.

Figure 1 shows the total correlation functions, $T(r)$, obtained by the Fourier transformation of $S(Q)$s for xLi-(1-x)Si (x=0, 0.1, 0.3, 0.5) amorphous samples with Q range from 0.4 to 35 $Å^{-1}$. Sharp peak was observed at 2.4 Å in $T(r)$ for the a-Si and it was attributed to Si-Si nearest-neighbor correlation. Si-Si correlation peaks were also observed in $T(r)$ for the Li-Si amorphous alloys, and they slightly shifted to a higher r with increasing Li concentration. The results indicate that the framework structures of a-Si were modified by Li insertion. Additionally, broad peaks were observed at around 1.6 Å in $T(r)$ for every sample. These peaks were attributed to Si-O correlation in accordance with the structure of SiO_2 glass. Thus, this result suggests that the samples may be oxidized and the framework structures of xLi-(1-x)Si amorphous samples were constructed by Si-Si and Si-O correlations. On the other hand, a characteristic negative peak at 2.8 Å emerged in $T(r)$ for 0.5Li-0.5Si. The peak was assigned to the Li-Si correlation since the neutron scattering length of Li has a negative value. In the future, we will perform the detailed analysis for Si-Si, Si-O, and Li-Si correlation by combination with high energy X-ray diffraction data, and elucidate a relation between the structure and remarkable electrode properties.