

 <b>MLF Experimental Report</b>	提出日 Date of Report
課題番号 Project No. 2012B0238 実験課題名 Title of experiment Structure of sulfur/carbon composite material for an all-solid-state battery 実験責任者名 Name of principal investigator Miki NAGAO 所属 Affiliation KEK	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 2012/12/14

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.
Carbon nanotube x 2 Sulfur/carbon nanotube composite material

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.
Sulfur/carbon nanotube composite material synthesized from carbon nanotube is potential electrode material for Li-S all-solid-state batteries. In order to determine a local structural change (especially for in-plane structure) of sulfur/carbon nanotube composite material in the synthesis process, we carried out total neutron scattering experiment. The aim of this research is to clarify a reaction mechanism of Li-S all-solid-state batteries by using small-angle scattering, diffraction and total scattering data.

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

Acid-treated carbon nanotube (C\_acid900) was synthesized from carbon nanotube (C) by treatment with  $\text{H}_2\text{SO}_4$  and  $\text{HNO}_3$ , and washed, dried under vacuum and heated at 900 °C. S/C\_acid900 composite sample was synthesized by gas phase mixing method.

The neutron total scattering experiments were carried out with the High Intensity Total Diffractometer (NOVA), installed at BL21 of MLF in the J-PARC. C, C\_acid900 and S/C\_acid900 were put into a cylindrical vanadium-nickel cell (6 mm in diameter) under air. The neutron total scattering data of the samples were measured for 4 hours at room temperature. Structure factor,  $S(Q)$ , of all samples were observed up to high  $Q$  region ( $Q = 68 \text{ \AA}^{-1}$ ) and converged to 1. Fig. 1 shows pair distribution function,  $g(r)$ , obtained by the Fourier transformation of  $S(Q)$ s for the samples with  $Q$  range from 0.5 to  $68 \text{ \AA}^{-1}$ . The first, second and third peak in  $g(r)$  for every sample is attributed to C-C correlation in structure of six-membered ring for graphene layer. The peak positions of  $g(r)$  for C observed at 1.42, 2.46 and  $2.84 \text{ \AA}$  correspond to that of a six-membered ring without strain as graphite. Fourth peak position indicates a curvature factor for graphene layer because the peak is assigned to the C-C correlation for neighboring six-membered ring. Compared to  $g(r)$  for C, there were no change in the peak positions of C\_acid900 and S/C\_acid900. This result indicates that the in-plane structure of carbon nanotube for framework of positive electrode are kept even if the acid treatment and sulfur mixing.

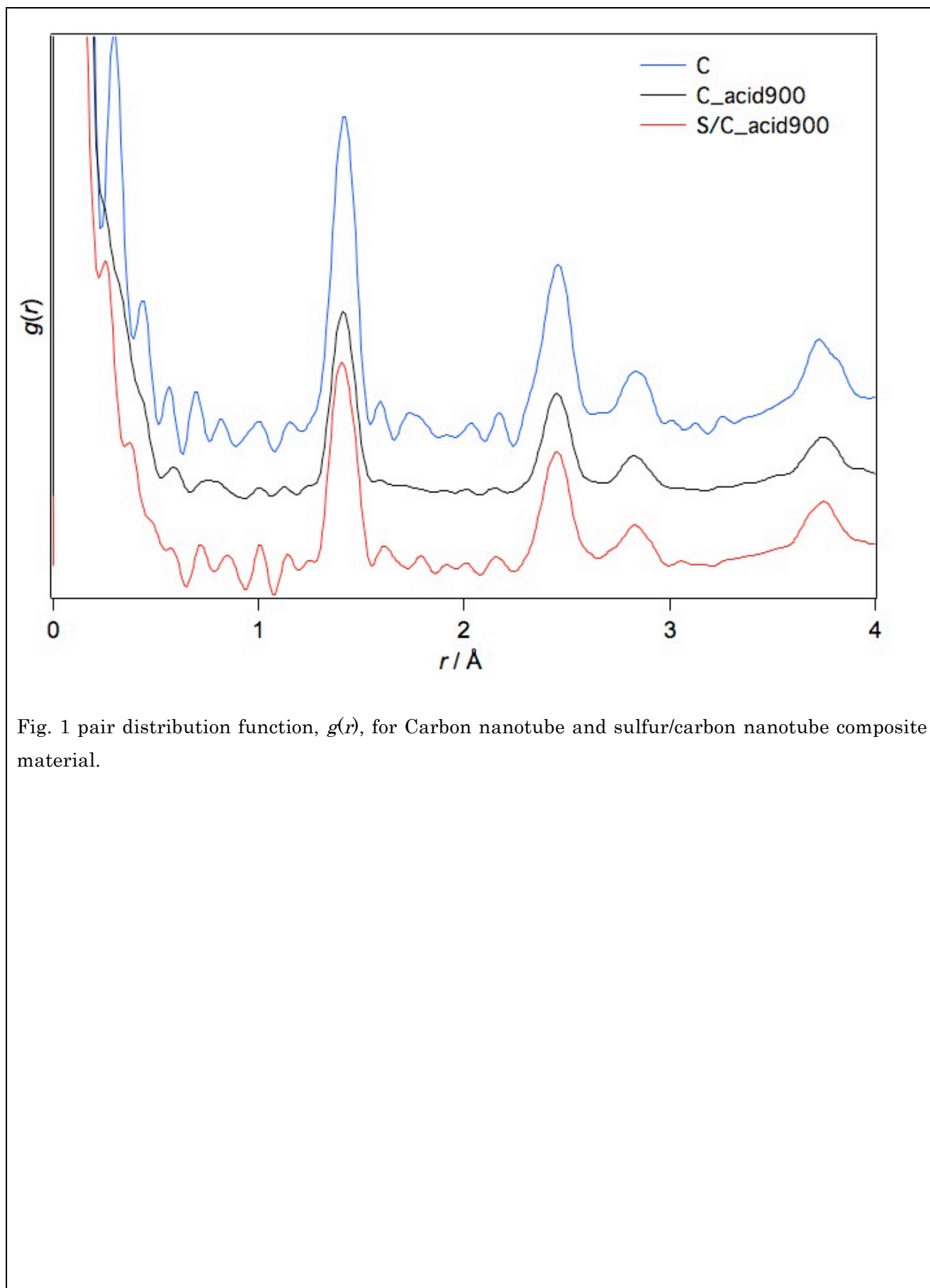


Fig. 1 pair distribution function,  $g(r)$ , for Carbon nanotube and sulfur/carbon nanotube composite material.