MLF Experimental Report

課題番号 Project No. 2014A0325	提出日 Date of Report 2014/8/27
実験課題名 Title of experiment Measurement of g(r,t) of the protonic conductor Rb3H(SeO4)2	装置責任者 Name of responsible person Kenji Nakajima
実験責任者名 Name of principal investigator Ryoji Kiyanagi	装置名 Name of Instrument/(BL No.) AMATERAS (BL14)
所属 Affiliation J-PARC center	実施日 Date of Experiment 2014/6/18 – 2014/6/23

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.

Rb3H(SeO4)2, powder, 5g

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.

$Rb_3H(SeO_4)_2$ exhibits superprotonic conductivity above Tc = 449 K. The protons are considered to migrate inside the material through disordered hydrogen bonds, but the details have not been well understood, yet. The purpose of this experiment is to elucidate the dynamic behavior of the conducting protons in the superprotonic phase of $Rb_3H(SeO_4)_2$ by examining g(r,t) derived from observed S(Q,ω).

The powder sample of $Rb_3H(SeO_4)_2$ was sealed in a vanadium can and attached to a sample stick with a heater. The sample stick was loaded on a cryostat at BL14. The measurements were performed in the temperature range between 300 K to 480 K with two different chopper settings; a low resolution (LR) mode and a high resolution (HR) mode.

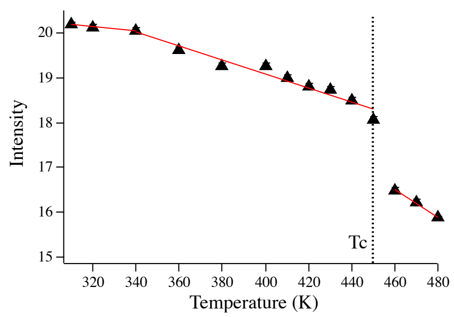

Fig. 1 Variation of the elastic intensities with respect to temperature.

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

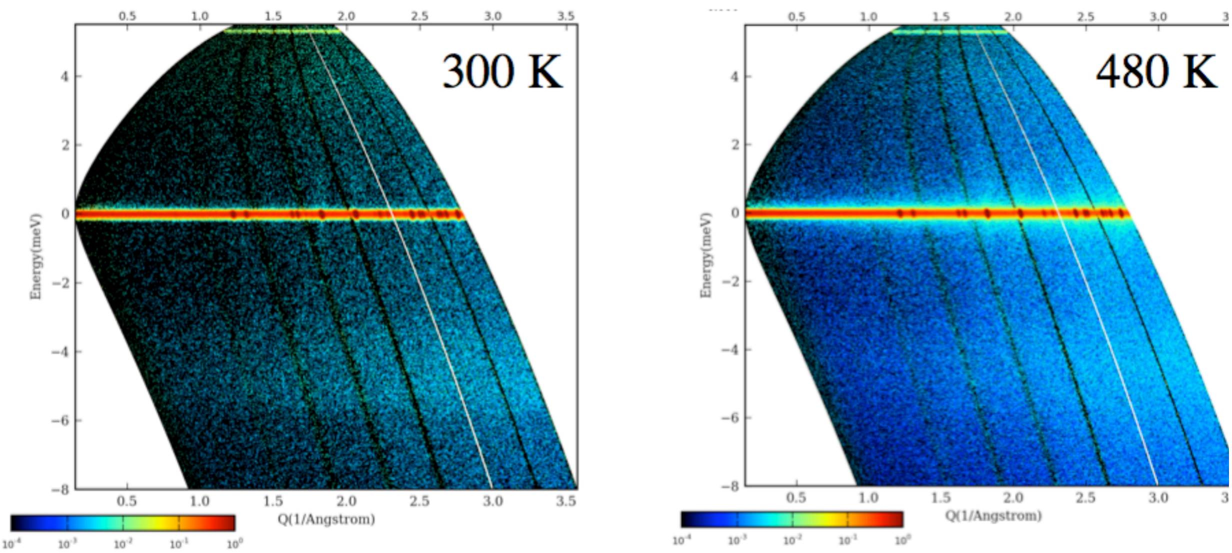

Fig. 2 Observed S(Q,w) at 300 K (left) and 480 K(right).

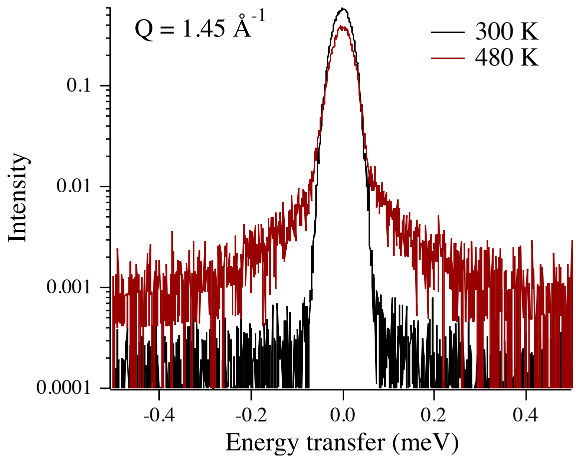

Fig. 3 Comparison of the quasi-elastic signals between 300 K and 480 K

After the tuning of the choppers, at first, quick scans, so-called "elastic scans", were performed with the LR mode as a function of temperature in order to confirm the phase transition temperature in the actual experimental setting. Figure 1 shows the variation of the elastic intensities with respect to the temperature. It is easily noticed that there is a gap around 450 K in the intensity, which clearly indicates the phase transition of the system to the superprotonic phase. In addition, a small kink can be seen around 340 K. This may indicate that the protons become diffusive even much below Tc. These observations well agree with previous NMR studies.

As next measurements, inelastic signals, that is S(Q,ω), were measured below and above the phase transition temperature. The measurements were performed with the LR and the HR modes to cover wider Q range and gain a fine resolution in Q-space. 6 hours were spent for each measurement in order to achieve good statistics. Figure 2 shows the observed S(Q,ω) at 300 K and 480 K with the incident neutron energy of 5.9 meV and Fig. 3 shows one dimensional profiles in the quasi-elastic energy region. As is clearly seen, significant quasi-elastic signals were recognized in the S(Q,ω) at 480 K, which indicates the conductive motion of the protons. In addition, a broad peak was observed around 5 meV especially in higher Q region. The transformation of the observed S(Q,ω) to g(r,t) will give much deeper insights about the proton motion, and the analyses are under way.