

 <b>MLF Experimental Report</b>	提出日 Date of Report 20/4/2013
課題番号 Project No. 2012B0225 実験課題名 Title of experiment Neutron diffraction study of sintering reaction processes for superconducting materials 実験責任者名 Name of principal investigator Yoshinori Tsuchiya 所属 Affiliation National Institute for Materials Science	装置責任者 Name of responsible person Kazuya Aizawa Stefanus Harjo 装置名 Name of Instrument/(BL No.) TAKUMI/(BL 19) 実施日 Date of Experiment 21/12/2012-23/12/2012 1/2/2013-4/2/2013

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.
Samples: Ag-sheathed (Bi,Pb) <sub>2</sub> Sr <sub>2</sub> Ca <sub>2</sub> Cu <sub>3</sub> O <sub>x</sub> monofilament tape (Bi,Pb-2223/Ag) Ag-sheathed (Bi,Pb) <sub>2</sub> Sr <sub>2</sub> CaCu <sub>2</sub> O <sub>x</sub> monofilament tape (Bi,Pb-2212/Ag) Physical form: Tape (4 x 40 x 0.3 mm <sup>3</sup> )

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.
1. Neutron diffraction measurements at room temperature for heat-treated samples Heat-treated BSCCO/Ag tapes have been prepared in advance under various conditions of gas atmosphere. The gas atmosphere of heat-treatments was mixture of nitrogen and oxygen. A starting state of a superconducting layer of the sample was a Bi2212 phase. Concentration of the oxygen was set to 2 to 25% and pressure was controlled in 1 to 3 atm. An annealing temperature was set to 830 C and an annealing time was 12 hours. The neutron diffraction measurements were performed at room temperature. The annealed samples were set perpendicularly and the tape surface and applied to 45 degrees to an incident beam. An exposure time was about 2 hours for one sample. Figure 1 shows the diffraction patterns of the heat-treated samples that annealed in 1 atm for various oxygen concentrations. In the heat-treatment condition of 1 atm and 12 hours annealing, it was found that a Bi2223 phase was appeared at the oxygen concentration of 2 to 20 % as shown in fig. 1 by open circles. Especially, diffraction peaks of the Bi2223 phase were appeared strongly in the sample annealed in oxygen

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

concentration of 5 and 10%. The diffraction peaks of the Bi2212 phase were relatively decreased for these annealing conditions.

### 2. In-situ neutron diffraction measurements during heat-treatment for various oxygen concentration and gas pressure

In order to observe a phase transition process in the heat-treatment of an actual superconducting wire, we performed an *in-situ* neutron diffraction measurement installing a gold-image furnace on a sample stage of the TAKUMI diffractometer. Starting sample was an untreated Bi2212/Ag tape and the sample was put into a platinum cell of the furnace. The annealing temperature was set to 830 C. In this study, three attempts were made in different heat-treating atmosphere. The heat-treatment process was about 6 hours for one measurement. A diffraction data was corrected as an event data in the whole measurement run, and the data was treated by a time-sharing process.

Figure 2 shows time-sharing neutron diffraction spectra of BSCCO/Ag tape during the annealing process of 830 C. The atmosphere was 1 atm and 5% of oxygen concentration. The spectra were integrated in every 15 minutes. As shown in fig. 2, peak intensities of the Bi2223 phase increased while that of the Bi2212 phase decreased with the annealing time. Namely, the phase transition behavior of Bi-superconducting phase was observed through a sheath material for an actual wire.

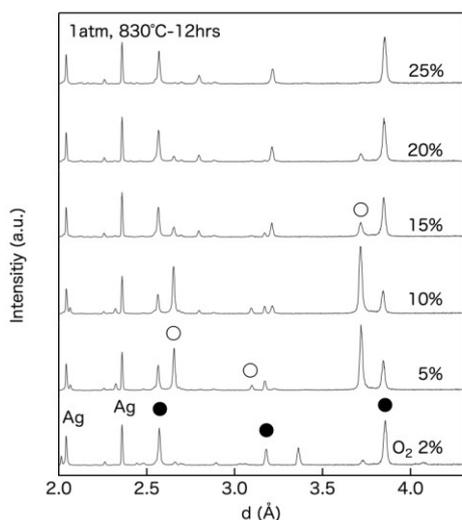


Fig.1 Neutron diffraction patterns of heat-treated BSCCO/Ag tapes under various oxygen concentrations at 830C. Filled circles indicate Bi2212 phase while open circles show Bi2223.

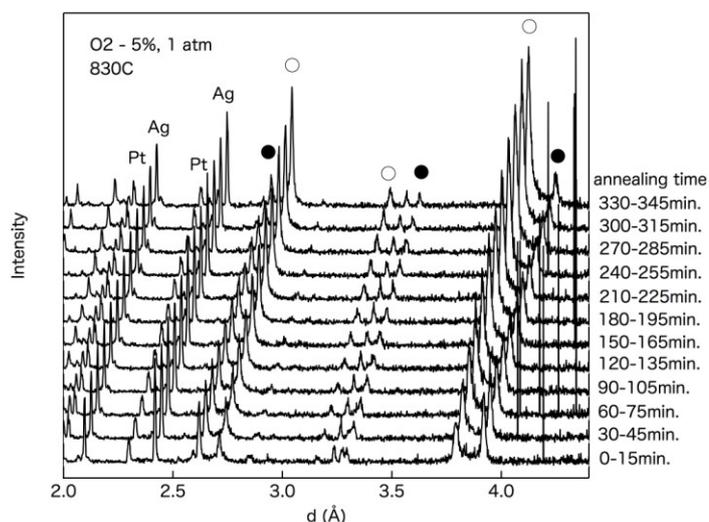


Fig.2 Change of neutron diffraction patterns during heat-treatment process of BSCCO/Ag tape. Filled circles indicate Bi2212 phase while open circles show Bi2223.