MLF Experimental Report	提出日 Date of Report 4/9/2012
課題番号 Project No. 2012A0092 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 14/5/2012-16/5/2012 15/6/2012-17/6/2012

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.

Specimens: Ag-sheathed $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ monofilament tape (Bi,Pb-2223/Ag)
Ag-sheathed $(Bi,Pb)_2Sr_2CaCu_2O_x$ monofilament tape (Bi,Pb-2212/Ag)
Specimen shape: Tape (4 x 20 x 0.2 mm^3), 5 tapes were stacked

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:

Two types of high-T_c superconducting Bi2223/Ag tapes are supplied from a company, one of which is the product one and the other one without final heat treatment in the manufacture processes. The latter used mainly in this experiment consists of mostly a starting Bi2212 powder. To observe the phase transition from Bi2212 to Bi2223 and its dependence on the condition of heat treatment process, we have designed newly a gold image furnace capable of high-pressure oxygen atmosphere and performed time-sharing neutron diffraction at BL19.

The rolling direction of the stacked Bi2212/Ag tape specimen was set vertically into the furnace. In order to measure the diffraction patterns for both the surface and the cross directions simultaneously, the surface of the specimen was inclined at 45 degrees to the incident neutron beam. The specimen was heated in N_2-O_2 gaseous mixture. The mixture ratio of O_2 was controlled 10 to 20% and the gas pressure was set in 1 to 3 atm. The elevated temperature process of room temperature to 1103K was 3 hours and the temperature was held for 3 hours. The diffraction data was treated using a time-sharing process. Pre-annealed specimens for long time treatment were also measured at room temperature.

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

Results:

In the beginning of this experiment, we verified operation of the newly designed furnace and its availability to the TAKUMI diffractometer. Relatively low background in a neutron diffraction pattern was achieved thanks to well designed furnace and collimators. Since the grain size of the starting Bi2212 powder in the specimen was very fine, the diffraction pattern of the as-received (non-reacted) specimen showed very few peaks excepting for peaks of Ag sheath at room temperature. In the heating process, the diffraction peaks of both Bi2212 and Bi2223 phase appeared above about 900 K (fig. 1). By comparing the diffraction patterns between the north and the south bank of neutron counters of the TAKUMI diffractometer, a strong crystal orientation was observed against a rolling direction of the specimen (fig. 2). Namely, the high oriented structure of the Bi2223/Ag superconductive wire is formed at an early stage of annealing process. The growth of peaks of Bi2223 was found to be very slow. Thus, the annealing process for 3 hours, which is the appropriately maximum time in the limited allocated machine time, is not enough to observe all the reaction process of the whole Bi2223 phase in the tape. For effective use of the limited machine time, we have also prepared pre-annealed specimens under various conditions changing the time of the heat treatment, pressure and the mixture ratio of O_2 and N_2. It has been revealed that a generation of impurities was suppressed for the annealed specimen under 3 atm atmosphere compared with that for 1 atm atmosphere (fig. 3). This clearly indicates that high pressure of the atmosphere is effective for an improvement of the quality of the Bi2223 tape. We are continuing this subject with more various heat treatment conditions, especially with higher pressures of the atmosphere.

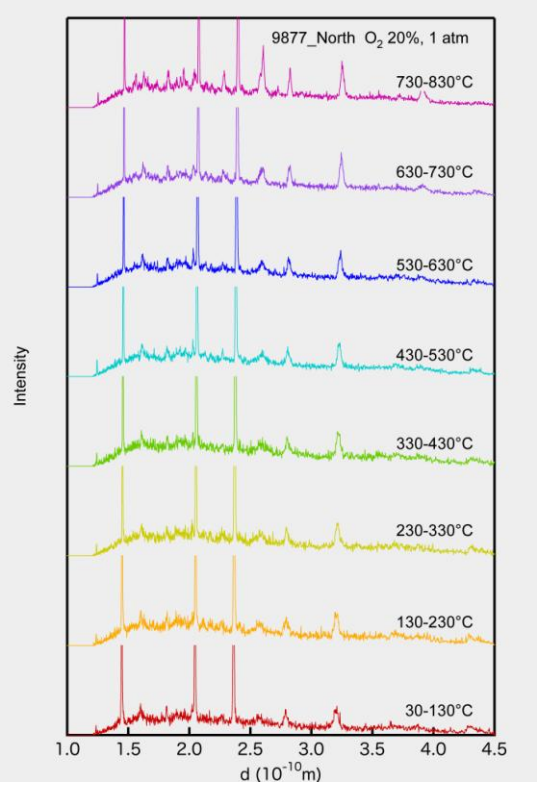

Fig. 1 Neutron diffraction spectra of the Bi2212/Ag tape for several temperature ranges during the heating process.

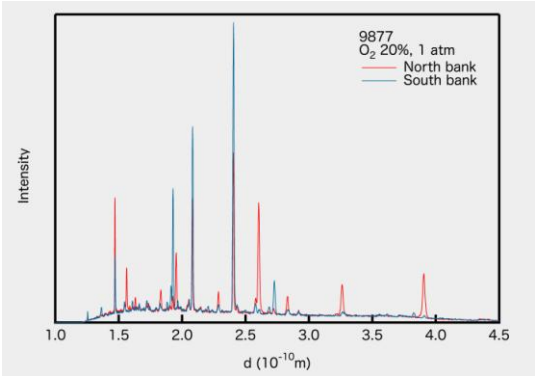

Fig. 2 Diffraction spectra for thickness and width directions of the tape specimen.

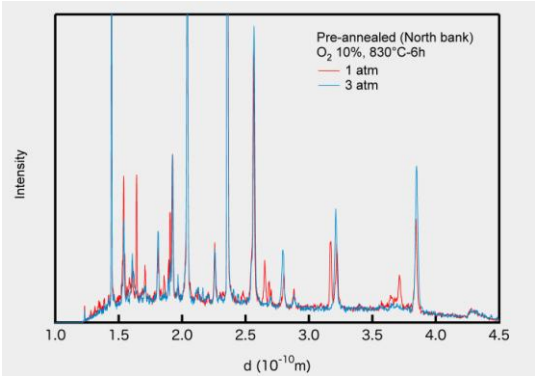

Fig. 3 Difference of the diffraction patterns by conditions of annealing pressure.