

 MLF Experimental Report	提出日 Date of Report 28/4/2011
課題番号 Project No. 2010A0085 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/(BL19) 実施日 Date of Experiment 18/10/2010-19/10/2010 23/11/2010-25/11/2010

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) ₂ Sr ₂ Ca ₂ Cu ₃ O _x monofilament tape (Bi,Pb-2223/Ag) Ag-sheathed (Bi,Pb) ₂ Sr ₂ Ca ₁ Cu ₂ O _x monofilament tape (Bi,Pb-2212/Ag) Specimen shape: Tape (4 x 20 x 0.2 mm ³), 10 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: Change of diffraction pattern of the Bi,Pb-2223 phase on Ag-sheathed (Bi,Pb) ₂ Sr ₂ Ca ₂ Cu ₃ O _x monofilament tape (Bi,Pb-2223/Ag) in annealing process between room temperature and 1023 K. Furthermore, arising of diffraction pattern of the Bi,Pb-2223 phase was observed on Ag-sheathed (Bi,Pb) ₂ Sr ₂ CaCu ₂ O _x monofilament tape (Bi,Pb-2212/Ag) in heating process between room temperature and 1118 K. Since, the tape sample has <i>c</i> -axis orientation to the surface of the tape, the change of the diffraction pattern of the sample by the orientation was observed using wide objective counter of TAKUMI diffractometer simultaneously. For these measurements, ten tape samples were stacked and put into a gold image furnace. The axial direction of sample was arranged to 45 degrees against the incident beam line. In this arrangement, the signal from the <i>c</i> -plane of Bi,Pb-2223 phase could be detected the south bank detectors of the TAKUMI diffractometer. The width of the incident beam was set to 5 mm. The collimation of sample-to-detector was open. The neutron diffraction measurements were performed at several temperatures up to 1023 K. Each measurement time was about 2 hours. The environment of the sample was Ar gas flow condition during heating.

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

Results:

Figure 1 shows diffraction pattern of the Bi,Pb-2223/Ag tape at room temperature. The data acquisition time was about 2 hours. In those patterns, the background spectra by the furnace were subtracted. The south bank detector detected the c plane of the Bi,Pb-2223 phase, and the other hands, the north bank detected the orthogonal a and b plane, clearly. The diffraction peaks of Ag sheath were also detected. In the measurement, it was found that the superconducting phase of the sample could be detected through the Ag sheath layers. Figure 2 and 3 show the diffraction patterns of Bi,Pb-2223/Ag tape by north and south banks at several temperatures. The temperature was kept for 2 hours by step during the diffraction pattern acquisition. In these measurements, a decomposition of the Bi,Pb-2223 phase did not observe at even 1023 K.

Later in this measurement, to observe phase transition of Bi,Pb-2212 phase to Bi,Pb-2223 during annealing, the neutron diffraction measurements were performed on the unreacted Bi,Pb-2212/Ag tape specimen at room temperature and heating process to 1118 K using a gold image furnace. At room temperature, the diffraction peak of Bi,Pb-2212 phase did not appear on the both detector bank. The reason is unknown. But there were a possibility to appear the final phase i.e. Bi,Pb-2223 superconducting phase by annealing. Industrially, the Bi,Pb-2223 phase should be obtained by annealing Bi,Pb-2212/Ag tape at 1118 K in high pressure of oxygen atmosphere for 8 to 30 hours. Referring the industrial process, we performed the neutron diffraction measurement at 1118 K in Ag gas flow condition for about 24 hours in total. However, the Bi,Pb-2223 phase did not appear.

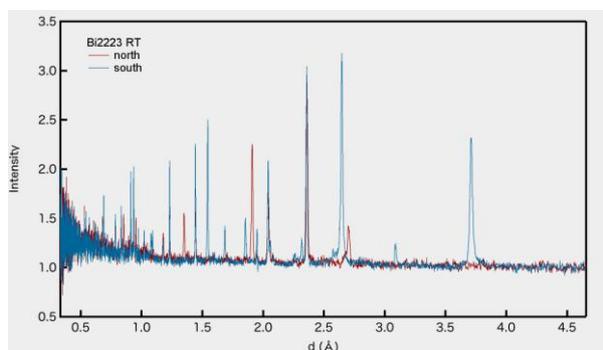


Figure 1: The diffraction pattern of the Ag sheathed Bi,Pb-2223/Ag tape at room temperature.

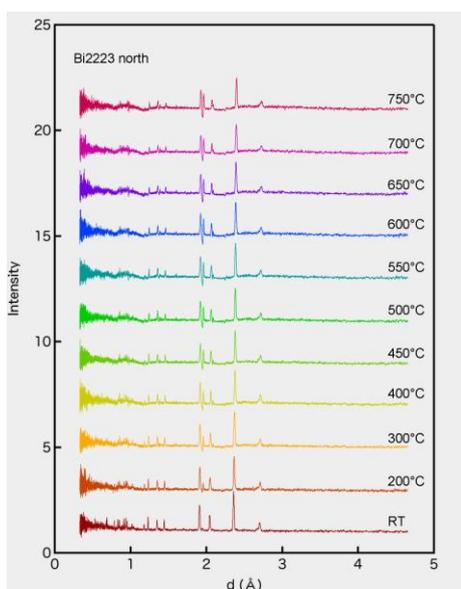


Figure 2: The diffraction patterns of Bi,Pb-2223/Ag tape by the north bank at several temperatures.

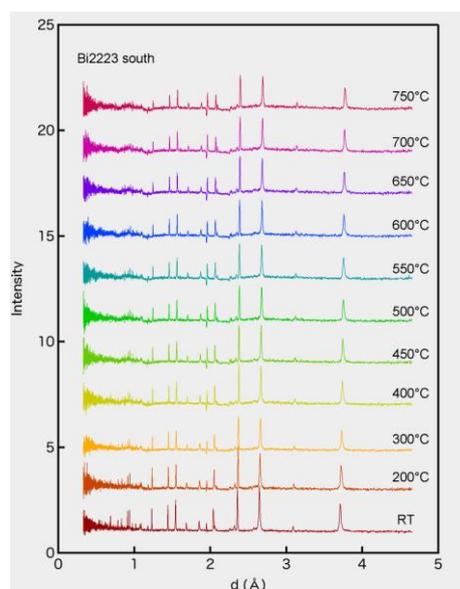


Figure 3: The diffraction patterns of Bi,Pb-2223/Ag tape by the south bank at several temperatures.