MLF Experimental Report	提出日 Date of Report 11/6/30
課題番号 Project No. 2009A0009 実験課題名 Title of experiment Crystal and magnetic structure of multiferroic BiFeO3-BaTiO3 composite 実験責任者名 Name of principal investigator KIMURA, Hiroyuki 所属 Affiliation IMRAM Tohoku university	装置責任者 Name of responsible person KAMIYAMA, takashi 装置名 Name of Instrument/(BL No.) SHRPD/BL08 実施日 Date of Experiment 10/05/21-10/05/23

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.

Solid solution of $(1-x)BiFeO_3+xBaTiO_3$ (x = 0.1, 0.25, 0.33)

All samples are in powder form.

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.

The main purpose of this study is to examine the magnetic reflections arising from the very long period modulated magnetic structure of $(1-x)BiFeO_3+xBaTiO_3$ (BFO-BTO). Since the period is extremely long, a very high resolution is demanded to clearly observe the magnetic reflections. In order to achieve the requirement, the chopper was operated with a 10 Hz mode in order to reach a low Q region with the highest angle bank that can provide the highest resolution. The samples were sealed in vanadium cans in ambient atmosphere, and the cans were attached onto a room temperature sample changer. All measurements were conducted at room temperature. Each measurement required about 15 hours for the data collection to achieve the satisfactory statistics. The measured powder diffraction patterns are shown in Fig. 1.

The obtained diffraction patterns on SHRPD clearly exhibit its high resolution compared with the one obtained on HERMES. As for the peaks around d = 2.3 Å, two peaks are clearly separated in the patterns of SHRPD, which greatly helps to recognize the existence of a possible second phase. The second phase was identified with the combination of other experimental results as is described later.

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

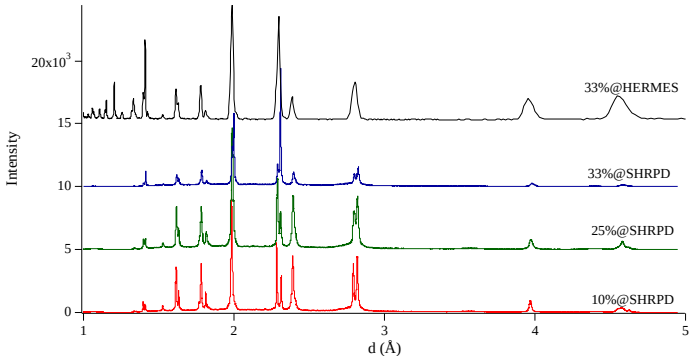

Fig. 1 The obtained powder diffraction patterns of (1-x)BFO-xBTO (x = 0.1, 0.25, 0.33). As a reference, the powder diffraction pattern measured on another powder diffractometer, HERMES at JRR-3M, is also shown.

The magnetic super reflections were clearly observed as shown in Fig. 2 (left). The peaks shown are (δ δ 3) series at d = 4.63 Å, that are extinct with a normal G−type antiferromagnetic structure, and (1+ δ δ 1) series around d = 4.555 Å. The (1+ δ δ 1) series reflections are heavily overlapped with each other because of a very small δ. However the broad peak can be well broken down to three peaks. The peak positions were well coincide with the previous reported ones confirming that the magnetic structure is modulated G−type antiferromagnetic with the very long period. The variations of the peak positions are shown in Fig. 2 (right). As is seen, the positions barely change regardless of the composition *x*.

The same compounds have been also studied by neutron and X−ray powder structure analyses. The analyses revealed that the original hexagonal phase of BFO coexists with a cubic phase in 0.2 > x >0.7, which is consistent with the powder pattern obtained on SHRPD (Fig. 1). At the same time, the composition of each phase was found to differ from each other. Ba and Ti ions were found to prefer to be incorporated in the cubic phase rather than the hexagonal phase. As a result, the actual composition in the hexagonal phase scarcely differs with respect to the nominal composition *x*. Besides, the relaxation of the rhombohedral distortion of the hexagonal structure was observed as the nominal composition x increases. Together with the result obtained in this experiment, it can be conjectured that the long period modulated magnetic structure is insensitive to the internal crystal structure.

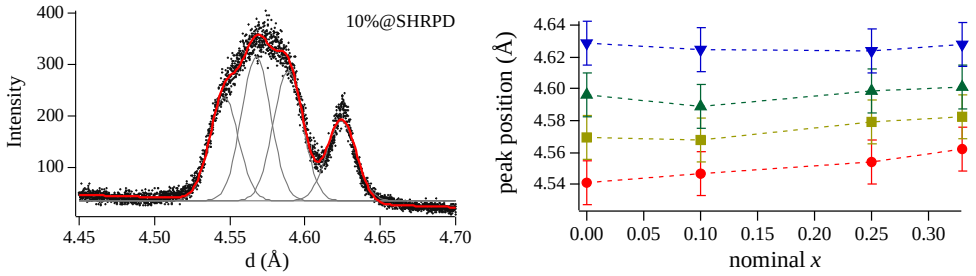

Fig. 2 The magnetic reflections and the variation of the their peak positions with respect to the concentration of x.