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Determination of crystal structure of tetragonal $PbFeO_2F$

Tetsuhiro Katsumata[1*], Yoshiyuki Inguma[1], Takashi Ida[2], Takao Turui[3]
[1]Gakushuin Univ., 1-5-1 Mejiro,Toshima-ku, Tokyo 155-0031, Japan
[2]Ceramics Research Laboratory, Nagoya Institute of Technology, Asahigaoka, Tajimi 507-0071, Japan
[3]Institute of Material Research, Tohoku University, Katahira, 2-1-1, Aoba-ku, Sedai, 980-8577, Japan

Introduction

Multi-ferroic materials, in which the ferroelectricity and ferromagnetism are present, have received renewed interest in recent years. The perovskite-type oxyfluoride, $PbFeO_2F$ is considered to be a candidate multiferroic material, because this compound includes A-site ion having a 6s lone pair and a 3d transition metal ion as B-site ion. Our group has been interested in this compound and investigated the structure and properties.[1,2]

$PbFeO_2F$ was synthesized under high pressure and high temperature.[3] The structure is a cubic perovskite-type structure in which the Pb ion shifts from the ideal A-site position along the twelve <110> directions.[1] The magnetic structure is G-type antiferromagnetic and the Neel temperature is 655(5) K.[2] Furthermore, it was revealed that the cubic $PbFeO_2F$ transforms to the tetragonal structure by annealing at 523-573 K and this tetragonal $PbFeO_2F$ was found to undergo a reversible phase transition to cubic in the vicinity of 475 K.[2] However, the crystal structure of the tetragonal $PbFeO_2F$ has not been cleared yet.

Here, we performed the synchrotron X-ray powder diffraction and the electron diffraction for the tetragonal $PbFeO_2F$ and tried to determine its crystal structure.

Experimental

Cubic $PbFeO_2F$ was synthesized under high pressure and high temperature. PbF_2, PbO, Fe_2O_3 TiO_2 were used as starting materials. Well ground mixtures of these materials were dried by evacuating at approximately 200 °C for one night. The mixed powders were reacted at 4-6 GPa and 1000 °C for 30 min. The tetragonal $PbFeO_2F$ is prepared by annealing the cubic $PbFeO_2F$ at 573 K for 3 weeks under Ar atmosphere. Synchrotron X-ray powder diffraction data were collected using the multi-detector system installed at the beam line BL-4B of Photon Factory, KEK, Tsukuba. The wave length was determined to be λ=1.206278(7)Å. The HRTEM observation was performed with a JEOL JEM-4000EX microscope.

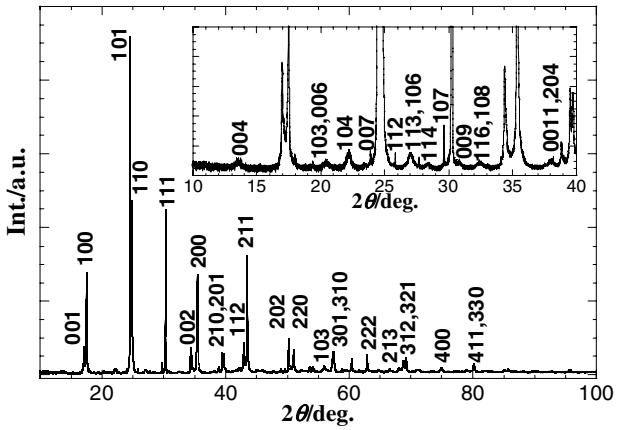

Figure 1 X-ray diffraction pattern for tetragonal $PbFeO_2F$. The peaks in the main figure and inset were indexed by $a\times a\times c$ and $a\times a\times 5c$ cells, respectively.

Results and discussion

Figure 1 shows the X-ray diffraction patterns for the tetragonal $PbFeO_2F$. Note that the many weak peaks were observed, as shown in the inset of figure 1. These peaks disappeared above 475 K with the structural change, thus these are considered to be related to a super structure of the tetragonal phase.

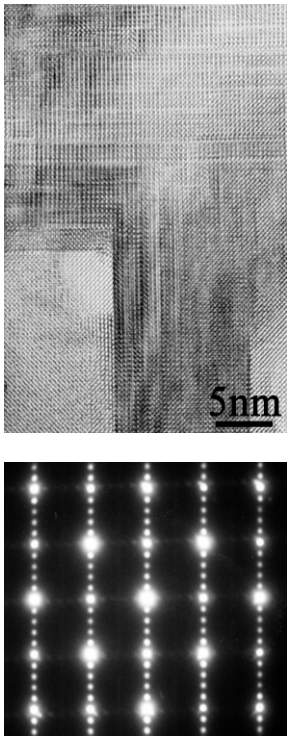

Figure 2 An HRTEM image and the electron diffraction pattern for tetragonal $PbFeO_2F$.

To determine the unit cell of the tetragonal $PbFeO_2F$, the HRTEM observation and the electron diffraction were carried out. Figure 2 shows the HRTEM image and the electron diffraction pattern for the tetragonal $PbFeO_2F$. As shown in the upper figure, the 5×1 superstructure and 90° oriented domain were observed. In the electron diffraction pattern, the (0 0 1/5) superlattice reflections were clearly observed. Therefore, the tetragonal $PbFeO_2F$ is found to have $a\times a\times 5c$ super structure. In fact, the weak X-ray peaks could be indexed on the $a\times a\times 5c$ multiple cell. The determination of the crystal structure is in progress now.

References

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*e-mail:20001996@gakushuin.ac.jp