Crystallography

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Relationship between the angular resolution of powder diffractometer and the accuracy of transition temperature in lanthanum titanate perovskite $La_{0.63}(Ti_{0.92},Nb_{0.08})O_3$

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Introduction

Doped $La_{2/3}TiO_3$ compounds have layered perovskite-type structure and exhibit excellent electrical and dielectric properties. The precision of unit-cell parameters is improved by higher-resolution diffractometer. Therefore the accuracy of the transition point would also be improved by using higher-resolution instrument. However, to our best knowledge, the effect of angular resolution on the accuracy of a transition temperature has not been investigated in the literature. Here, we have studied the temperature dependence of unit-cell parameters of $La_{0.63}(Nb_{0.92},Nb_{0.08})O_3$ by three diffractometers with different angular resolutions, to investigate the relationship between the accuracy of the transition temperature and the angular resolution. The details are described in our paper (M. Yashima *et al.*, *Chem. Phys. Lett.*, **363** (2003) 582).

Experiments

The $La_{0.63}(Ti_{0.92},Nb_{0.08})O_3$ compound was prepared by solid-state reactions. We have investigated *in situ* a continuous transition between the orthorhombic and tetragonal phases in $La_{0.63}(Ti_{0.92},Nb_{0.08})O_3$ by three X-ray powder diffractometers with different $\Delta d/d$ resolutions of 0.03%, 0.06% and 0.10%. The d and Δd denote the $\lambda/(2\cdot\sin\theta)$ and peak width where λ and θ are wavelength of X-ray and Bragg angle. For the low-resolution experiments, a conventional Cu $K\alpha$ X-ray diffractometer was used. The intermediate- and high-resolution experiments were done at the beam line 3A of the Photon Factory, KEK, Japan.

Results and discussion

The maximum temperature where the peak splitting between 400 and 040 reflections is detectable T_{max} were 225˚C, 306˚C and 339˚C for the low-, intermediate- and high-resolution diffraction experiments, respectively. The transition temperatures T_c determined by the power law were estimated to be 456˚C, 396˚C and 360˚C for low-, intermediate-, and high-resolution data, respectively (Fig. 1). Thus, the T_{max} increases, while the T_c decreases with decreasing of $\Delta d/d$ value. The accuracy ΔT_c was ±2˚C, ±14˚C, and ±228˚C for $\Delta d/d$ resolutions of 0.03%, 0.06% and 0.10%, respectively (Fig. 1). Only the highest-resolution diffractometer of $\Delta d/d$ =0.03% was able to detect the peak splitting between 400 and 040 reflections between 327˚C and 339˚C. The high resolution of $\Delta d/d$=0.03% is required to determine the transition point with the precision of ±2˚C. The present powder diffraction work would give useful information for the studies of phase transition and for many applications in crystallography and materials science.

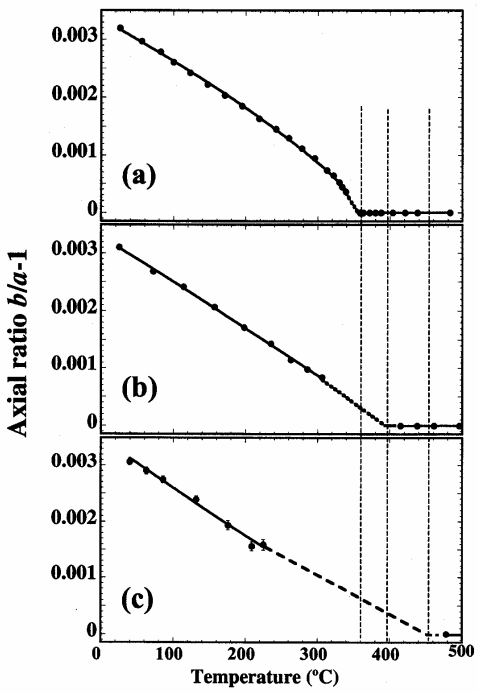

Fig.1. Temperature dependence of the axial ratio b/a-1 determined by (a) high-, (b) intermediate-, and (c) low-resolution diffractometers. Solid lines are least-squares fits to the experimental data by the power law. Dashed lines are extrapolations calculated by the optimized parameters.

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