Study of structural phase transition of 10 mol %Nb-doped $La_{2/3}TiO_3$ monitored by synchrotron X-ray power diffraction

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Introduction

Doped $La_{2/3}TiO_3$ compounds have perovskite structure and exhibit electrical and dielectrical properties. Thermal expansion and temperature dependence of lattice parameters are important factors in designing the components of solid oxide fuel cells. Conventional X-ray diffractometer produces double wavelengths of $K\alpha_1$+$K\alpha_2$, and broad diffraction peaks with asymmetric shape, which lead inaccuracy in determining the peak positions and thus lattice parameters. Here, we have used synchrotron X-ray diffraction technique, having higher angular resolution and simple peak shape (no $K\alpha_2$ peak) compared with conventional X-ray diffractometry, to determine precise lattice parameters of $La_{0.633}(Ti_{0.90}Nb_{0.10})O_3$ as a function of temperature and to investigate of phase transition temperature.

Experiments

The synchrotron X-ray diffraction data were collected using beam line BL-3A installed at Photon Factory, KEK, Tsukuba. The wavelength was determined to be λ=1.37873(3) Å after the calibration with NIST CeO_2 sample (a=5.41129 Å). Experimental conditions were: step interval = 0.01 , counting time = 4 s, 2θ range from 40.35° to 41.95°. Peak positions were determined using a profile fitting program *PRO-FIT* (Toraya 1986).

Results and discussion

Peak splitting between orthorhombic 020 and 200 reflections was clearly observed at lower temperatures but it merged at higher temperatures (Fig. 1). With increasing temperature 020 and 200 peaks approached and became single between 349° and 370°C. Although near the transition temperature these two peaks seemed single, but up to 349°C the profile (020 and 200) could be resolved successfully.

Temperature dependence of lattice parameters is shown in Fig.2. With increasing temperature, a and c/2 parameters increased considerably compared with b parameter, resulting that a and b parameters coincided around 370°C where the orthorhombic phase transformed to tetragonal symmetry. (1) The lattice parameters increased continuously, while b/a ratio decreased continuously with temperature and became unity at the orthorhombic-tetragonal transition point. (2) Good agreement was obtained in the lattice parameter values between heating and cooling. These results of (1) and (2) suggest that the orthorhombic-tetragonal phase transition is of second order.

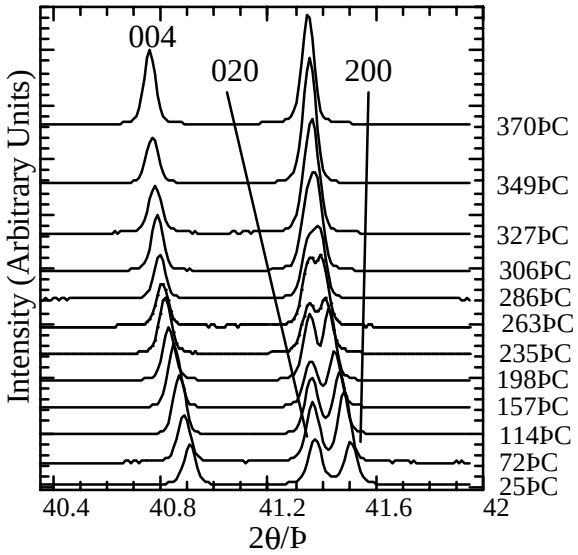

Fig.1. Temperature dependence of 004, 020 and 200 peak profiles of $La_{0.633}(Ti_{0.90}Nb_{0.10})O_3$.

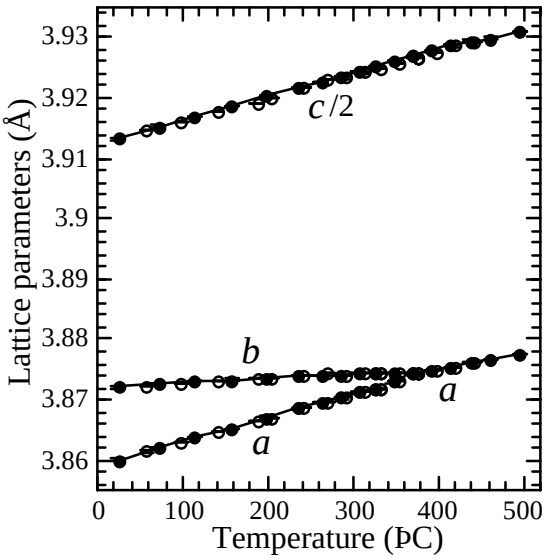

Fig.2. Temperature dependence of lattice parameters of $La_{0.633}(Ti_{0.90}Nb_{0.10})O_3$. Fill and open circles denote heating and cooling data, respectively.

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