Phase Change of $Zr_{0.868}Y_{0.132}O_{1.934}$ during Annealing at 1000 °C

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Introduction

Zirconia-based ceramics have fluorite-related phases with relatively simple crystal structures, but the phase transitions are poorly understood. One of the most important keys to solve this problem is the study of the diffusionless cubic-tetragonal (*c*-*t'*) phase transition where *t'* emphasizes a metastable tetragonal phase. Thus, *t'* can be distinguished from the stable *t* which formed diffusionally. The synchrotron X-ray diffraction is a powerful tool to investigate the *c*-*t'* phase change [1]. We have investigated the *c*-*t'* phase change of arc-melted zirconia and hafnia solid solutions by using synchrotron X-ray diffraction. But, from the view point of industrial applications, sintered materials are more important than arc-melted samples. Here, we report the *c*-*t'* phase change of sintered $Zr_{0.868}Y_{0.132}O_{1.934}$ samples during annealing at 1000ûC using synchrotron X-ray powder diffraction.

Experiments

$Zr_{0.868}Y_{0.132}O_{1.934}$ solid solutions (Dai-Ichi-Kigenso-Kagaku Co.) were annealed at 1000°C for 0 to 999 h after sintering at 1650°C for 4 h. The intensity data were collected using the synchrotron radiation (λ=1.38 Å) and the triple-axis/four-circle diffractometer as a powder diffractometer at the beam line BL-3A of Photon Factory, KEK, Tsukuba. The scanning conditions were: 2θ range from 63.000û to 66.000û and $\Delta 2\theta = 0.005°$.

Results and discussion

Figure 1 shows the synchrotron X-ray powder diffraction profiles of the $Zr_{0.868}Y_{0.132}O_{1.934}$ samples annealed at 1000°C for 0 to 999 hours. The diffraction profile for the sample annealed at 1000°C for 0 h, exhibited an asymmetric peak with a shoulder at higher 2θ position. This peak profile can be decomposed into three peaks of $004_{t'}$ and $400_{t''\text{ and/or cubic}}$ and $400_{t'}$ reflections where *t"* is a tetragonal form with an axial ratio of unity [1, 2]. The $400_{t''\text{ and/or cubic}}$ peak intensity decreased, while the $004_{t'}$ and $400_{t'}$ intensities increased with an increase of annealing time at 1000ûC. This indicates that the fraction of *t'* form increased while that of *t"* and/or cubic decreased during the annealing. After annealing for 999 h at 1000°C, the *t"* and/or cubic peak disappeared. Thus, the *t"* and/or cubic form in the title compound prepared by sintering the commercial powders transformed into *t'* form during the annealing. Decrease in ion conductivity during annealing at 1000ûC is attributable to this transformation.

References

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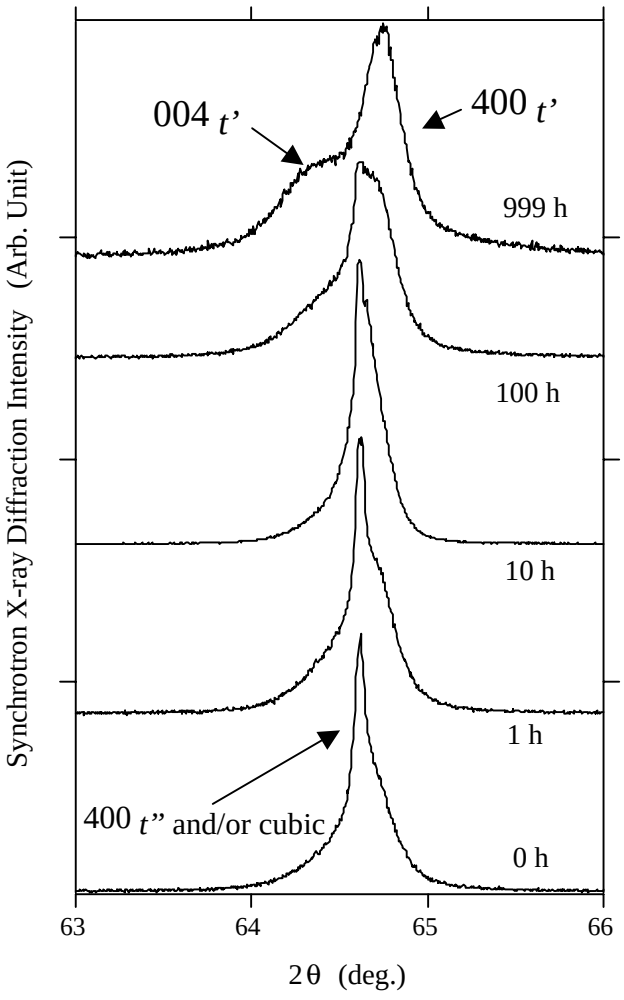

Fig. 1: Synchrotron X-ray diffraction profiles of the sample annealed at 1000°C for different periods of 0 to 999 hours.