Powder diffraction data on $Ca_{0.9}Nd_{0.1}Ti_{0.9}Al_{0.1}O_3$

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Single crystals of $Ca_{0.9}Nd_{0.1}Ti_{0.9}Al_{0.1}O_3$ (CNTAO) were grown using optical floating zone technique and the grown crystals were characterized by Laue diffraction and powder X-ray diffraction techniques for crystal quality and its composition, respectively. The powder pattern of CNTAO was indexed and refined using GSAS program to an orthorhombic structure with space group *Pbnm* (#62), $a = 5.3832(1)$, $b = 5.4343(1)$, $c = 7.6389(2)$ Å, $V = 223.4677$ $Å^{3'}$, and $Z = 4$. © *2015 International Centre for Diffraction Data.* [doi:10.1017/S0885715615000342]

Key words: $CaTiO_3$, single crystals, powder X-ray diffraction

I. INTRODUCTION

$CaTiO_3$, a well-known perovskite that crystallizes in orthorhombic structure with *Pbnm* space group is used as a major phase in synroc, which can immobilize rare earths and long-lived actinides (Ewing *et al.*, 2007), as dielectric resonators in wireless communication systems (Jancar *et al.*, 2003) and for phosphor materials (Lemanski *et al.*, 2011). The similar ionic radii of calcium (Ca^{2+}: 0.134 nm) with neodymium (Nd^{3+}: 0.127 nm) makes $CaTiO_3$ a suitable host for efficient strong red luminescence under UV excitation (Dereń *et al.*, 2008). About 70% of Ca^{2+} cations in $CaTiO_3$ could be replaced by Nd^{3+} cations and substitution of Al^{3+} in Ti^{4+} site for charge compensation does not affect the crystal structure (Kipkoech *et al.*, 2003). Owing to its potential applications, we have grown single crystals of $Ca_{0.9}Nd_{0.1}Ti_{0.9}Al_{0.1}O_3$ and its powder X-ray diffraction (PXRD) results are being reported here.

II. EXPERIMENTAL

A. Synthesis

Polycrystalline $Ca_{0.9}Nd_{0.1}Ti_{0.9}Al_{0.1}O_3$ (CNTAO) was prepared by solid-state reaction. Stoichiometric ratios of high purity (4N) powders of $CaCO_3$, Nd_2O_3, TiO_2, and Al_2O_3 were mixed by ball milling to obtain a homogenous powder of CNTAO. The mixture was calcined at 1200 °C for 10 h in air with intermediate grinding. PXRD was done on the sample to confirm the single phase, and after the confirmation, the powders were packed and sealed into a rubber tube which was evacuated using a vacuum pump. The powders were compacted in the form of rods using hydraulic press under an isostatic pressure of 70 MPa. These rods were densified by sintering at 1300 °C for 12 h in air.

Single crystals were grown using these rods as feed and seed rods in a four mirror optical floating zone furnace (Crystal Systems Corp. FZ-T-4000-H-HR-I-VPO-PC). Counter rotations of 20–30 rpm of the feed and seed rods and a translation of 10–20 mm h^{-1} in argon atmosphere resulted in good quality crystals. The grown single crystals were crushed and ground in an agate mortar and pestle to particle sizes of ~10 µm for compositional characterization.

B. Data collection

PXRD patterns of crushed CNTAO single-crystal powders were recorded at room temperature using a STOE X-ray powder diffractometer operated in Bragg–Brentano geometry with fixed slits. The diffraction data were recorded using CuK_α radiation operated at 40 kV and 30 mA. The 2θ scan range was from 21° to 87° with a step size of 0.05° and a count time of 40 s $step^{-1}$. The powder was loaded in a zero background (911) Si single-crystal wafer holder.

III. RESULTS

Experimental powder diffraction pattern (symbols) corresponding to CNTAO powder is displayed in Figure 1. Initial structure solution was obtained using Index and Refine

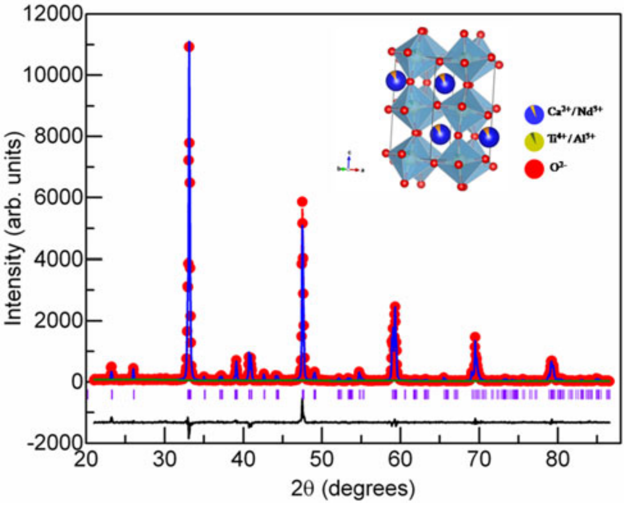

Figure 1. (Color online) The crystal structure of CNTAO drawn by Vesta software (Momma and Izumi, 2013).

[a)] Author to whom correspondence should be addressed. Electronic mail: nithya@igcar.gov.in subroutine in WinXPOW software available with STOE diffractomer. Then the pattern was indexed and the cell parameters were refined with *Pbnm* space group. Also, Rietveld refinement of the whole powder diffraction pattern was performed using GSAS program (Larson and Von Dreele, 2000). Background intensity was fitted using a linear interpolation function (solid line–green color). Diffracted peaks were adequately fitted using Pseudo-Voigt function. The calculated powder pattern is also included in the same Figure 1 (solid line – blue color). Below the diffraction pattern, difference between the calculated and experimental patterns (solid line – black) is shown. Vertical lines (pink color) shown at the bottom in the figure represent the expected Bragg diffraction peaks as per the space group (*Pbnm*) used for refinement. The cell parameters obtained from both refinements are in good agreement.

Inset in Figure 1 illustrates the crystal structure of CNTAO drawn by Vesta software (Momma and Izumi, 2013).

IV. CONCLUSION

The CNTAO structure has been refined. The compound shows an orthorhombic distorted-perovskite structure. PXRD data have been generated for this composition which can be included into the PDF database as a new entry.

SUPPLEMENTARY MATERIALS AND METHODS

The Supplementary material referred to in this article can be found online at journals.cambridge.org/pdj.

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