Structural Studies on a Complex Calcium Niobate (75.25:24.75, $CaO:Nb_2O_5$) Based on the Perovskite Structure

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Complex niobates are of potential interest as dielectric ceramics for use in wireless communications. In an effort to clarify structural inconsistencies reported in $Ca_3Nb_2O_8$, we are studying the CaO-Al_2O_3-Nb_2O_5 system. In our re-examination of the CaO-rich section of the CaO-Nb_2O_5 system, we discovered that the perovskite-related structure at a composition of "75.25:24.75, $CaO:Nb_2O_5$" is more complex than was previously reported. In order to determine the structure of this material, several data sets have been collected including: single crystal X-ray precession photographs, single crystal data collected on a four-circle diffractometer, single crystal data obtained using a CCD area detector, conventional X-ray powder diffraction data, X-ray powder diffraction data with a position sensitive detector and $K\alpha 2$ removed, and powder neutron diffraction data. The single crystal X-ray precession patterns of "75.25:24.75, $CaO:Nb_2O_5$" can be completely accounted for by a cubic unit cell with a = 23.934 Å and superstructure reflections showing 6 times the basic perovskite unit cell. Refinements of the single crystal and powder neutron diffraction data in cubic, tetragonal, and rhombohedral symmetries result in R_{wp} ~ 20%. Optical microscopy indicates that the crystals are weakly birefringent rather than being isotropic and show no signs of twinning. In an attempt to refine the crystal structure, high-resolution synchrotron X-ray powder diffraction data were collected (λ = 0.70019 Å). These data indicate that several strong peaks are split into two peaks, indicating deviation from higher symmetry. Efforts are being made to solve the structure in a C-centered monoclinic setting of the cell.