Poster Presentations

[MS24-P11] Molecular-to-Material Pathway: A Preparation of Ba-Nb Oxides from Metal-organic Framework.

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A novel compound $\{Ba_2(H_2O)_5[NbO(C_2O_4)_3]HC_2O_4\}.H_2O$ (1) has been prepared and characterized by single-crystal XRD, IR and thermal analysis. The ability of 1 to act as a single-source precursor for the formation of bimetallic oxides has been explored by thermal analysis and XRPD. The thermal treatment of 1 at the chosen temperature (600-1200 °C) followed by cooling, results in the formation of the mixed-metal $Ba_5Nb_4O_{15}$ and/or $Ba_4Nb_2O_9$ oxide phases. The $Ba_5Nb_4O_{15}$, as the major crystalline oxide phase, forms at ~700 °C. Three stable $Ba_4Nb_2O_9$ polymorphs have been isolated: already well known hexagonal α-form [1-3] and orthorhombic γ-form [1-3], and so far unknown hexagonal δ-polymorph, having reduced symmetry 6H-perovskite structure. Heating 1 at 1135 °C and then cooling to RT leads to formation of α-$Ba_4Nb_2O_9$, while the same procedure at 1175 °C results in the crystallization of another two polymorphs, γ-$Ba_4Nb_2O_9$ and δ-$Ba_4Nb_2O_9$. Electrical measurements were performed on samples prepared by pelleting milled single crystals of 1 and heated to chosen temperatures. The results of conductivity measurements were completely comparable with those reported for $Ba_4Nb_2O_9$ ceramics prepared via multiple reheating steps, typical for conventional ceramics preparations [3]. Therefore, we believe that proposed synthetic procedure deserves additional attention since the benefit of "two in one" approach - one-step preparation of the desired oxide phase which is already in a form suitable for conductivity measurements - was successfully established. This study also focuses on controlling the phase composition and the crystallite domain lengths by altering preparation conditions, namely: (i) the time for which samples were held at the given temperature and (ii) the cooling rate. High temperature γ-$Ba_4Nb_2O_9$ polymorph has been successfully retained and stabilized at room temperature (RT); desired crystallite size in nanoscale regime, ranging from ~5 to 20 nm, can easily be tuned. The crystallite domain length and lattice strain were calculated from X-ray diffraction (XRD) line broadening analysis performed during the Rietveld structure refinement.

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