

Thermal Stability and Electrical Conductivity of $\text{Ca}_5\text{Mg}_{4-x}\text{M}_x(\text{VO}_4)_6$ ($0 \leq x \leq 4$) where $\text{M} = \text{Zn}, \text{Ni}$ Measured by Impedance Spectroscopy

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Abstract : Calcium oxovanadates with garnet related structure are multifunctional oxides in various fields like photoluminescence, microwave dielectrics, and magneto-dielectrics. For example, vanadate garnets are self-luminescent compounds. They attract attention as RE-free broadband excitation and emission phosphors and are candidate materials for UV-based white light-emitting diodes (WLEDs). $\text{Ca}_5\text{M}_4(\text{VO}_4)_6$ ($\text{M} = \text{Mg}, \text{Zn}, \text{Co}, \text{Ni}, \text{Mn}$) compounds are also considered promising for application in microwave devices as substrate materials. However, the relation between their structure, composition and physical/chemical properties remains unclear. Given the above-listed observations, goals of this study are to synthesise $\text{Ca}_5\text{M}_4(\text{VO}_4)_6$ ($\text{M} = \text{Mg}, \text{Zn}, \text{Ni}$) and to study their thermal and electrical properties. Solid solutions $\text{Ca}_5\text{Mg}_{4-x}\text{M}_x(\text{VO}_4)_6$ ($0 \leq x \leq 4$) where M is Zn and Ni have been synthesized by sol-gel method. The single-phase character of the final products was checked by powder X-ray diffraction on a Rigaku D/MAX-2200 X-ray diffractometer using $\text{Cu K}\alpha$ radiation in the 2θ range from 15° to 70° . The dependence of thermal properties on chemical composition of solid solutions was studied using simultaneous thermal analyses (DSC and TG). Thermal analyses were conducted in a Netzch simultaneous analyser STA 449C Jupiter, in Ar atmosphere, in temperature range from 25 to 1100°C heat rate was $10 \text{ K}\cdot\text{min}^{-1}$. Coefficients of thermal expansion (CTE) were obtained by dilatometry measurements in air up to 800°C using a Netzsch 402PC dilatometer; heat rate was $1 \text{ K}\cdot\text{min}^{-1}$. Impedance spectra were obtained via the two-probe technique with an impedance meter Parstat 2273 in air up to 700°C with the variation of pH_2O from 0.04 to 3.35 kPa. Cation deficiency in Ca and Mg sublattice under the substitution of MgO with ZnO up to 1/6 was observed using Rietveld refinement of the crystal structure. Melting point was found to decrease with x changing from 0 to 4 in $\text{Ca}_5\text{Mg}_{4-x}\text{M}_x(\text{VO}_4)_6$ where M is Zn and Ni . It was observed that electrical conductivity does not depend on air humidity. The reported study was funded by the RFBR Grant No. 17-03-01280. Sample attestation was carried out in the Shared Access Centers at the IHTE UB RAS.

Keywords : garnet structure, electrical conductivity, thermal expansion, thermal properties

Conference Title : ICSSCN 2018 : International Conference on Solid State Chemistry and Nanosciences

Conference Location : Tokyo, Japan

Conference Dates : March 27-28, 2018