INVESTIGATIONS ON SYNTHESIS, MICROSTRUCTURAL, CRYSTALLOGRAPHIC, SPECTROSCOPIC AND THERMAL CHARACTERISTICS OF RANDOM PYROCHLORE AND PEROVSKITE CERAMICS

ABSTRACT

Ceramic materials are inorganic and non-metallic compounds with high fusion temperature, exhibiting poor conductivity and high oxidation resistance. The ceramic materials are a direct result of their internal structure. The ability to control structures through processing and develop new structures through various techniques requires the qualitative and quantitative analysis of the atomic and electronic structures.These materials' physicochemical and mechanical properties are purely dependent on the crystal structure, lattice defect, exposed lattice plane, surface morphology, and particle sizes.Owing to the potential applications in various fields, there is demand to synthesize Perovskite and Pyrochlore structured materials with improved properties. According to literature, alkaline earth zirconates belong to family of oxides with general formula $A^{2+}B^{4+}O_3$ exhibit Perovskite type structures and $A_2^{2+}B_2^{4+}O_7$ exhibiting Pyrochlore structures. ABO_3 Perovskites doped with acceptor-ions show proton conductivity at high temperatures, making them a potential candidate for electrochemical devices.$A_2B_2O_7$ type materials are generaly used for catalyst, Encapsulation, sensors, SOFC , Batteries, Radiation absorption, Hosts for nuclear wastes, thermal barrier coatings, ferroelectric and multiferroelectrics.

In the present work, synthesis of pure perovskites of $CaZrO_3$,$BaZrO_3$,$SrZrO_3$ (zirconates) ,a potential pyrochlore compound $Ce_2Zr_2O_7$ (Zirconate) and hybrid phase possessing compound $LaAlO_3$-$La_2Zr_2O_7$ (LAZ) were synthesized using sol-gel assisted combustion synthesis using nitrate-based precusors.Alkaline earth zirconate hosts are known for their outstanding properties such as high chemical and thermal stability, single phase crystalline structure, high refractive index, and wide band gap, making them perfect for electronic applications ceramics, gas sensors, optical coatings, and filters. Rare earth zirconates have low thermal expansion coefficient values and high fracture toughness relating to a toughned ceramic systems. $Ce_2Zr_2O_7$ are also believed to contribute to grain bridging and Improved mechanical properties in air-sintered cerium zirconatecomposites.In general, pyrochlores have a high melting point, a relatively high coefficient of thermal expansion, and low thermal conductivity, making them suitable for high temperatures.Compound $LaAlO_3$-$La_2Zr_2O_7$ was prepared using a solgel assisted combustion method to find good attention for the material usage at the high temperatures and exhibit good fracture toughness in ceramics.

TG-DSC analysis showed the transition involving various intermediate reactions from which the phases formed for each sample were found. Different exothermic and endothermic reactions along with the temperature of formation were determined. Weight losses during each transformation were seen during gel to final product evolution.XRD analysis of all the synthesized compounds confirmed the presence of the respective pure phases (in the case of $CaZrO_3$, $BaZrO_3$, $SrZrO_3$, $Ce_2Zr_2O_7$, and LAZ) while additional partially transformed phases were found along with the dominant phases (in the case of LAZ and $BaZrO_3$). Theoretical calculations of various parameters such as lattice strain, crystallite size, strain-induced and dislocation density for each compound were determined using XRD data of compounds.UsingSPuDS theoretical calculation, two plots depicting the influence of temperature on various parameters such as global instability index, tolerance factor, lattice parameter, and bond valence sum of cations were constructed. From which the effect of the variation of temperature on other parameters was found out. From this, the influence of temperature on crystallographic parameters was determined.SEM analysis has shown that particle size ranges in microns and particle agglomeration were seen in all the compounds due to weak molecular attraction forces due to the wet synthesis method. TEM analysis was performed for all compounds, in which the particle morphology was analyzed. While the HRTEM images showed the lattice spacing matching with the results of XRD data and SAED analysis confirmed the crystal structure of compounds with a determined zone axis comparing the XRD data. EDS analysis confirmed the presence of elements along with their respective weight% and atomic%. Absorption bands due to metal-oxygen bonds and other reactions in the powder samples were determined by FT-IR spectroscopy analysis. Two absorption bands corresponding to tetrahedral and octahedral cations were identified for each sample. Force constants were theoretically calculated for all the compounds and the results were correlated.