

MICROWAVE COMBUSTION SYNTHESIS OF LEAD LANTHANUM TITANATE (Pb,Ln)TiO₃

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ABSTRACT

Microwave-assisted process is a technique used for the fast and controlled processing of advanced ceramic materials. Microwave heating process is fundamentally different from other heating processes. In the microwave process, heat is generated internally within the material instead of originating from external heating sources [1,2]. The advantages of microwave process are (1) significant reductions in manufacturing costs due to energy savings and shorter processing times; (2) improved product uniformity and yields; (3) improved microstructure and properties of the material [3,4]. Many novel methods such as spray pyrolysis, sol-gel, co precipitation and combustion techniques have been adopted for the synthesis of ferroelectric materials. Combustion synthesis process is a well-established technique for materials development and recently gaining much more importance for advanced materials [5,6]. Lead titanate (PT) is a very interesting ferroelectric material with perovskite type structure (ABO₃). Doped PT ceramics can show excellent pyroelectric and piezoelectric properties [7]. The microwave combustion is a synthesis process which makes use of the advantages of the microwave assisted techniques as volumetric and uniform heating, and the characteristics of the combustion process, as fast and low temperature synthesis. This work has as aim study the influence of lead oxide excess in microwave synthesis and sintering of lead lanthanum titanate perovskite oxide (PLT). The reaction of fuel with the metallic precursors yields an intermediate compound that decomposes exothermically providing high temperature conditions for the formation of the complex oxide. Due to the relatively high temperatures achieved on the combustion process (in this case, approximately 500 °C) and the possibility of lead evaporation in its oxide form, batch formulas were prepared with different amounts of excess of lead oxide by considering a (Pb,Ln)TiO₃ final stoichiometry with charge compensation vacancies in the A or B sites. The powders were isostatically pressed in cylindrical bodies with approximately 10mm of diameter and 4 mm of thickness. The samples were conventionally and microwave sintered. The microwave sintering was conducted in a 2.45 GHz equipment. The X-ray diffraction analysis indicated the fully formation of the perovskite (Pb,Ln)TiO₃ phase and also the presence of different amounts of the PbO in the submicrometric ceramic powders. The influence of the amount of the PbO excess was markedly observed in the densification process. From these results it was possible to predict the optimal batch formula for this synthesis procedure. The microstructure analysis was conducted by SEM. Based on the results was observed a more uniform microstructure in the microwave sintered samples.

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