Influence of the Structure of Titano-Zirconate Precursors on PZT Properties during Microwave Hydrothermal Process

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Abstract

PZT powders are usually prepared by conventional mixed oxide method using PbO, TiO2, and ZrO2. This process requires relatively high calcination and sintering temperatures in order to ensure a complete formation of the perovskite phase and an enhanced densification of the ceramic. So, the chemical processes are a more interesting alternative techniques of synthesis in term of reactivity and chemical homogeneity. The synthesized powders are fine and non-agglomerated at lower temperature. In this study, a fast synthesis of fully crystallized PZT is presented in a less basic medium (KOH 1M) and an excess of lead nitrate (30%) by microwave assisted hydrothermal method. We have developed two routes from two different heterobimetallic alkoxide derivatives of titanium and zirconium: alkoxide and amino-alkoxide routes involve respectively synthesis of a usual isopropoxide derivative (A) and a novel triethylamino oxide derivative (B). Our objective was to observe the supposed influence of the organometallic precursor on the mechanism of germination during microwave heating. So, both precursors had to present great differences of structure and chemical properties. At one hand, we have compared PZT powders characteristics obtained from both routes. Very pure crystalline phases (relative density of 100%) were prepared at 210°C (45 minutes) instead of 265°C (2 hours) by conventional heating. At the other hand, corresponding PZT disks were prepared at 1200°C and polarized to observe electrical properties. We have observed that the mechanisms of crystallization strongly depend on the structure of the precursor. All the powders were characterized by X-ray diffraction, scanning electron microscopy, laser grain sizes distribution and pycnometry. Homogeneous PZT ceramics were obtained by sintering the powders at 1200°C with 100°C below the conventional temperature and higher apparent density (about 98%). Comparing to materials synthesized by the traditional methods, a higher mechanical quality value and a smaller room temperature dissipation factor were noted.