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ROLE OF TEMPERATURE IN MICROSTRUCTURE AND PIEZOELECTRICAL PROPERTIES IN $Ba_{0.9}Ca_{0.1}Ti_{0.9}Zr_{0.1}O_3$ COMPOUND

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$Ba_{0.9}Ca_{0.1}Ti_{0.9}Zr_{0.1}O_3$ Lead free perovskite was prepared by the Pechini method. The resin obtained was dried at 60°C during 12 h. This precursor was analyzed by the X-ray diffraction (XRD) showing an amorphous phase. This one was heated at 700°C during 1h in order to stabilize the tetragonal crystal phase with P4mm symmetry. The resulting powders heated at 1200°C during 5 h showing the coexistence of two crystal phases: tetragonal and rhombohedral (R3m). The XRD patterns were refined using Rietveld method through Fullprof software. The results obtained by the thermal analysis, Fourier transform infrared spectroscopy and Raman spectra are in agreement with those reported in the literature. The atomic force microscopy (AFM, MFP-3D Infinity) image for the sample heated at 700°C shows a homogeneous particle size and round shape distribution in the nanoscale range. Transmission electron microscopy analysis (TEM-Philips CM 200) shows large pieces of agglomerated of homogeneous round-shape particles. The average particle size was 30±1.4 nm. The elemental analysis in TEM-electron energy loss spectroscopy (EELS) mode were performed through the Zr $M_{2,3}$-edge, Ca $L_{2,3}$-edge, Ti $L_{2,3}$ edge, O K-edge and Ba $M_{4,5}$-edge. The ligand field multiplet theory was employed to confirm the presence of one oxidation state for Ti^{4+} at the $L_{2,3}$ edge in O_h symmetry with a crystal-field splitting about 2.15 eV. The field emission scanning electron microscopy (FESEM, JSM-7401F) analysis indicates the well-developed grains. The average grain size for the sample heated at 700°C was determined in the nanometer range in comparison with the sample sintered at 1200°C that shows 20.4±3.8μm. The chemical composition as revealed by energy dispersive X-ray analyzer (EDAX, Hitachi SU3500) is found to be near to stoichiometric ratio of the compound. The ceramics sintered at 1200°C for 5h exhibits a piezoelectric coefficient d_{33}=40pC/N; remanent polarization, P_r=7μC/cm^2 and coercive field, E_c =0.5kV/cm.

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