Low temperature sintering and dielectric properties of BaTiO₃ – type oxyfluorides

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Barium titanate is a well-known multifunctional ferroelectric mostly used as dielectric in the fabrication of multilayer ceramic capacitors (MLCCs). However, the sintering temperature ($T_{sint} \sim 1400$ °C) and the Curie temperature ($T_C \sim 120$ °C) of pure BaTiO₃ are too high for any industrial application at low factory cost. Furthermore, the dielectric permittivity peak (ϵ'_r) at T_C is very sharp and a slight variation of temperature induces a strong decrease of ϵ'_r . Therefore, since more than three decades, the research on barium titanate is mainly oriented towards the lowering of its sintering and Curie temperatures with the aid of fluorinated additives [1–5]. The aim of the present work is the preparation of BaTiO₃ bulk ceramics using barium and lithium fluorides.

Stoichiometric BaTiO₃ is first synthesized from BaCO₃ and TiO₂ (rutile). Several chemical compositions 0.97mol.BaTiO₃ + 0.03mol.BaF₂ + ymol.LiF (0.03 \leq y \leq 0.15) are prepared and wet-milled in ethanol. The powder mixtures are then cold-pressed to pellets and sintered in various conditions. The obtained ceramics are investigated by X-ray diffraction and scanning electron microscopy. Dielectric measurements are carried out at 1 kHz in the temperature range 200 – 500 K.

Each ceramic displays a broad maximum of permittivity at T_C which is around room temperature. The ceramic with initial composition 0.97 mol. BaTiO₃ + 0.03 mol. BaF₂ + 0.12 mol. LiF sintered at 950 °C for 2 h. exhibits the best performances: T_C ~ 10 °C, ε'_{max} = 5300, tan δ = 0.004. Such dielectric characteristics make this material of great interest for manufacturing cheap MLCCs. On the other hand, the bulk ceramic of this oxyfluoride could be used as target for thin films deposition in the manufacture of micro devices.

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