SIZE EFFECT OF $CaTiO_3$ PARTICLES ON THE DIELECTRIC PROPERTIES OF CERAMICS CONTAINING CaF_2 AND LiF

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The developments in computer memories and mobile telephones have accelerated the need for new technical ceramics with better performances to manufacture microchips at reduced costs. This demand has focused attention on perovskite materials and their solid solutions. For a long time, lead-based electroceramics have occupied the first place in the huge market of electronic components from medical imaging devices to diesel injector actuator. Today, the presence of Pb becomes a serious threat for human health and environment. Therefore, in the last decade, the research in material science has been directed towards the search of lead-free ceramics. In this way, calcium titanate and its solid solutions are intensively studied worldwide and could be promising candidates for MMICs (Microwave Monolithic Integrated Circuits). In an earliar work we studied the chemical system $(1-x)CaTiO_3 - x(CaF_2+LiF)$ and a solid solution was obtained in the initial composition range $0 \leq x \leq 0.30$. The present investigation reports the influence of $CaTiO_3$ particle size on the dielectric properties of ceramics sintered with the aid of a (CaF_2+LiF) mixture.

$CaTiO_3$ is first synthesized by solid state reaction between $CaCO_3$ and TiO_2 at 850 °C for 4 h. After cooling, the powder is re-ground and annealed at the same temperature for 2 h. Calcium titanate is then separated in several particle size classes. The particle size distribution is in the range 75 - 475 µm. For each class of particle size, ceramics are sintered at 950 °C for 4 h using 10 mol. % of (CaF_2+LiF) as additive. Dielectric measurements are performed on the samples at respectively 100 Hz and 1 kHz from room temperature to 500 °C under nitrogen gas (N_2). The thermal variation of the permitivity ε'_r and losses $\tan\delta$ display the same profile whatever the granulometry and the frequency are. Three dielectric anomalies are detected in the temperature range investigated. Between 25 °C and ~ 150 °C, ε'_r and $\tan\delta$ are very stable and frequency independent. Beyond 150 °C, ε'_r increases strongly whereas $\tan\delta$ exhibits a broad maximum around 350 °C varying in the range 25 - 60 with particle size. Upper 400 °C, giant values of the permitivity reaching 800000 are observed. When the frequency increases, the height of ε'_r anomalies decreases and $\tan\delta$ maximum increases shifting to higher temperatures. This behavior is characteristic of phase transitions with relaxation. All the ceramics are of interest in the fabrication of class I capacitors.

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