Preparation and characterization of new lead-free ceramics related to $CaTiO_3$

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Day after day, technological innovations are emerging rapidly and the search for new materials brings competitive advantages to industrial enterprises. Besides the understanding of the preparation process and properties, the novel materials require ecological impact and rational use of energy. To fill these conditions, today the trend is towards the development of lead-free ceramics sintered at low temperature. Actually, considerable attention is focused on $ATiO_3$-based ceramics (A = Ca, Sr or Ba).

Many years ago, we studied the effect of $KMgF_3$ on the sintering and dielectric properties of $BaTiO_3$, $KNbO_3$ and $NaNbO_3$ [1-3]. In the present work, we aim the investigation of $CaTiO_3$ ceramics chemically modified with the aid of potassium magnesium fluoride.

Calcium titanium oxide is synthesized in air by the conventional route at 850 °C. $KMgF_3$ is prepared at 700 °C in gold sealed tube. Various molar mixtures (1-x) $CaTiO_3$ – x $KMgF_3$ are dry-ground, pressed into pellets then sintered in ambient air at 900 °C for 2 h.

The obtained materials are analyzed by several techniques: X-ray diffraction (XRD); scanning electron microscopy (SEM); differential scanning calorimetry (DSC); thermogravimetry analysis (TGA, DTGA); dielectric measurements (DE).

As results, an oxifluoride solid solution with nominal composition $Ca_{1-x}K_xTi_{1-x}Mg_xO_{3-3x}F_{3x}$ occurs in the range $0 \leq x \leq 0.20$. The XRD spectra show the samples to have a distorted orthorhombic lattice of the perovskite structure at room temperature. The ceramic's grain size is between 0.5 µm and 5 µm. One or two second order phase transitions are detected by DE and confirmed by DSC (Table 1). These fluorinated dielectrics could be of interest to manufacture class I capacitors.

Table 1: DSC data of (1-x) $CaTiO_3$ – x $KMgF_3$ samples

Initial composition	T_1 (°C)	ΔH_1 (kJ.mol^{-1})	T_2 (°C)	ΔH_2 (kJ.mol^{-1})
$0.95CaTiO_3 – 0.05KMgF_3$	360	0.210	450	4.599
$0.90CaTiO_3 – 0.10KMgF_3$	295	1.585	375	0.407
$0.85CaTiO_3 – 0.15KMgF_3$	275	2.475	445	1.185
$0.90CaTiO_3 – 0.20KMgF_3$	370	0.925	-	-

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