Thermal stability of BST in a vast range of temperature

E. Morîntale¹, N.Scărişoreanu², M. Dinescu², P. Rotaru¹

¹University of Craiova, Faculty of Physics, 13 A.I. Cuza St., Craiova, RO-200585, Dolj, Romania
²INFLPR - National Institute for Laser, Plasma and Radiation Physics, PPAM - Lasers Department, 409 Atomistilor blvd., Magurele, RO-077125, Bucharest, Romania

Abstract

The paper describes the study of the thermal stability of two samples of barium strontium titanate (BST) with different contents of barium and strontium. The samples are thermal and thermogravimetric very stables.

Keywords: Ferroelectric oxides, BST, Thermal analysis

PACS numbers: 65.90.+i, 77.22.-d

1 BST structure and specific features

Ferroelectric oxides are essential components in a large number of applications, from ultrasound medical imaging [1]-[2] to nonvolatile random access memory [3] and micro-electromechanical systems [4]. Barium strontium titanate, BaₓSr₁₋ₓTiO₃ (BST) is a typical ferroelectric material suitable for microwave applications at room temperature [5]-[6]. The dielectric permittivity of BST sample has a maximum near the phase [5], [7]. The dependence of the dielectric permittivity of a homogeneous BST sample follows the Curie-Weiss law [7]. In agreement with this law the steepness of the temperature run of the dielectric permittivity must be two times higher to the left of the maximum (ferroelectric phase) than to the right of a maximum (paraelectric phase) [5]-[8].

Barium strontium titanate (BST), is a solid solution of BaTiO₃ with SrTiO₃. BaTiO₃ and Ba-rich BaₓSr₁₋ₓTiO₃ compounds have tetragonal perovskite structure Figure (1), [9], and are ferroelectric, while SrTiO₃ and Sr-rich BaₓSr₁₋ₓTiO₃ compounds have cubic perovskite structure Figure (2), [10], and are paraelectric at room temperature [11].

Lattice parameter of BST versus composition is show in Figure (3), [11]. Substituting Ba with Sr results in the decrease of the Curie temperature and the increase of the dielectric constant of the material [12]. The phase diagram of the BaₓTiO₃-SrTiO₃ system has been studied by several groups [13]-[17].

BST is used as nanofibers [18] and thin films [19]-[23]. For its applications is very important as the structure of the used material (BST) to be thermally stable up to very high temperatures. In this paper we will study the thermal stability of two samples of material: that BST40 where x = 0.4 and BST50 where x = 0.5 on a vast range of temperature.
Figure 1: $\text{Ba}_x\text{Sr}_{1-x}\text{TiO}_3$ tetragonal perovskite structure

Figure 2: $\text{Ba}_x\text{Sr}_{1-x}\text{TiO}_3$ cubic perovskite structure

Figure 3: Lattice parameter of $\text{Ba}_x\text{Sr}_{1-x}\text{TiO}_3$ vs. composition
2 Experimental

2.1 Preparation of barium strontium titanate (BST)

$\text{Ba}_{x}\text{Sr}_{1-x}\text{TiO}_3$ compounds with the molar formula $\text{Ba}_{0.4}\text{Sr}_{0.6}\text{TiO}_3$ and $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{TiO}_3$ were prepared by standard solid-state reaction [24]-[26]. The precursors used for preparing BST were barium carbonate ($\text{BaCO}_3$), strontium carbonate ($\text{SrCO}_3$) and titanium dioxide ($\text{TiO}_2$) powders of higher purity than 99.9 %. Mixing was carried out for 2h in an agate bottle containing agate balls [25]-[26]. The mixtures were calcined at 1100 °C for 2 hours, crushed, mixed with polyvinyl alcohol, and then pressed in a cylindrical mould to yield cylindrical pellets. BST samples were sintered in air at 1300 °C for 2 hours. The BST40 and BST50 samples with single phase cubic structure, good morphology and dielectric properties used as target for thin films deposition [25]-[26], are thermal analysed for determining thermal stability in air and nitrogen.

2.2 Experimental technique

Thermal analysis measurements (TG, DTG, DTA and DSC) of both BST40 and BST50 samples were carried out in air and nitrogen dynamic atmosphere (150 cm$^3$·min$^{-1}$ flow) under non-isothermal linear regime. A horizontal Diamond TG/DTG Analyzer from PerkinElmer Instruments was used during the measurements. Samples contained in $\text{Al}_2\text{O}_3$ crucibles, were heated from RT to 1200 °C with heating rate of 10 K·min$^{-1}$.

3 Results and discussion

Thermal analysis is a good technique in the study of the thermal stability of the compounds. This method was used in numerous papers [18], [24], [27]-[29].

Between thermal properties, thermal stability is particularly important when BST is used to deposition of thin films. We analyzed by thermal analysis two samples of ferroelectric materials: BST40 where $x = 0.4$ and BST50 where $x = 0.5$. The thermal analysis results of the both samples in air and nitrogen, at heating rate of 10 K·min$^{-1}$ to 1200 °C are shown in Figure (4) - (7).

The thermogravimetric results of BST40 and BST50 in air and nitrogen, at heating rate of 10 K·min$^{-1}$ to 1200 °C are shown in Table 1.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Gas</th>
<th>Mass (mg)</th>
<th>Mass loss (%)</th>
<th>Residual mass (mg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BST40</td>
<td>air</td>
<td>4.023</td>
<td>1.6</td>
<td>98.4</td>
</tr>
<tr>
<td></td>
<td>nitrogen</td>
<td>3.847</td>
<td>1.3</td>
<td>98.7</td>
</tr>
<tr>
<td>BST50</td>
<td>air</td>
<td>5.557</td>
<td>1.3</td>
<td>98.7</td>
</tr>
<tr>
<td></td>
<td>nitrogen</td>
<td>7.113</td>
<td>1.2</td>
<td>98.8</td>
</tr>
</tbody>
</table>
Figure 4: Thermal analysis results of BST40 in dynamic air at heating rate of 10 K·min⁻¹

Figure 5: Thermal analysis results of BST40 in dynamic nitrogen at heating rate of 10 K·min⁻¹
Figure 6: Thermal analysis results of BST50 in dynamic air at heating rate of 10 K min⁻¹.

Figure 7: Thermal analysis results of BST50 in dynamic nitrogen at heating rate of 10 K min⁻¹.
With increasing temperatures, little mass loss was observed below 430 °C for BST40 in air, and below 600 °C for BST40 in nitrogen. The same little mass loss was observed below 400 °C and up to 1000 °C for BST50 in air, and below 600 °C and up to 1000 °C for BST50 in nitrogen. On the other temperature ranges BST40 and BST50 are stables.

For DTA and DSC curves is observed that both samples have nearly constant heat capacity until 900 °C. Over 900 °C heat capacity increases almost linearly with temperature. To the entire temperature range RT-1200 °C not show thermal effects to be attributed to crystalline phase transformations.

4 Conclusions

Thermal analysis of two samples of ferroelectric material BST40 and BST50 confirmed good thermal stability of the studied samples. At decomposition in air and in nitrogen the samples have a weight loss of up to 1.6% at temperatures below 600 °C, which is due to desorption of water and gases physically and chemically adsorbed.

The BST50 sample has a minor mass loss at temperatures over 1000 °C can be attributed to incomplete calcinations of original material. The BST40 and BST50 samples are remarkably stable in terms of heat capacity and thermal effects.

References


