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Sintering behaviour and dielectric properties of $BaTiO_3$: Effect of borax addition

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Abstract

Barium titanate ($BaTiO_3$) is a prominent ferroelectric material widely utilized in various electronic applications, due to its excellent dielectric properties. Lowering its sintering temperature, a highly demanded requirement in multilayer ceramic capacitors (MLCC), enables the use of metal-based electrodes and consequently reduces the production cost. In the present work, we investigated the impact of using low melting glassy phases of borax ($Na_2B_4O_7 \cdot 10\,H_2O$) in developing low-temperature-sintered $BaTiO_3$ ceramics. The effects of borax addition on the densification, microstructure and dielectric properties of $BaTiO_3$ ceramics were investigated. Different amounts of borax were added to $BaTiO_3$ powders, and the compacts made from these mixtures were sintered at 1100 and 1150 °C for 3 h. The results revealed that this small addition of borax can effectively reduce the sintering temperature of $BaTiO_3$ to 1100 °C. The presence of the borax liquid phase at the $BaTiO_3$ grain boundaries enhanced densification at such low temperature, without formation of secondary phases or degradation of the dielectric properties. Relative permittivities up to 2410 and 7685 at room and Curie temperatures, respectively, were obtained.

Keywords: *$BaTiO_3$, sintering aid, liquid phase, densification, dielectric properties*

I. Introduction

Barium titanate ($BaTiO_3$ or BT) is a widely used electroceramics material known for its high permittivity, low dielectric loss, excellent piezoelectric properties [1,2] and remarkable electrocaloric strength [3]. Additionally, it exhibits outstanding optical and photoluminescence properties [4,5]. These unique properties make BT highly valuable and important for various electronic and engineering applications, including ceramic capacitors, multilayer ceramic capacitors (MLCCs), positive temperature coefficient (PTC) thermistors, piezoelectric devices [2,6], transducers [1], actuators [7] semiconductors [8], optical and optoelectronic devices [5] and solid-state cooling devices based on the electrocaloric effect (ECE) [9].

The sintering temperature plays a crucial role in determining the density, grain size, crystallinity and electrical properties of $BaTiO_3$ ceramics [10–13]. High-temperature sintering is essential to remove porosity and improve the material's density, accordingly leading to higher relative permittivity and reduced dielectric loss [10]. However, high sintering temperature necessitates the use of expensive noble metals for internal electrodes for MLCCs fabrication, which consequently increases the production costs of such an important application [14]. Therefore, lowering of the BT sintering temperature can eliminate the need for noble metal electrodes, making it more cost-effective while still maintaining desirable properties for MLCC production [15,16].

Previous studies have focused on optimizing densification and controlling grain growth in $BaTiO_3$ ceramics to improve their dielectric performance. This has been performed by using fine powder [10,12,17,18], specific sintering techniques [19,20] and liquid-phase additives [21–24]. Liquid-phase sintering, in particular, is a well-established technique that promotes consolidation at lower temperatures, offering a cost-effective way to enhance the sintering of industrial materials, such as cemented carbides and grinding tools [25]. The liquid-phase sintering process involves the formation of a reactive metastable liquid phase at the grain boundaries, which acts as a lubricant during the initial sintering

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stages. This phase facilitates particle rearrangement and grain boundary diffusion, leading to higher densification at lower temperatures [25,26]. Optimizing sintering parameters and dopant types and distribution can significantly improve the $BaTiO_3$ dielectric properties, by controlling the microstructure and ensuring a balanced interaction between donor and acceptor dopants [25,26].

Various sintering aids, including glasses, silicates, borates, fluorides and low-melting oxides have been used to enhance the sintering of $BaTiO_3$ material. While these compounds have proven effective, ongoing research seeks better sintering aids. In this study, we explored sodium borate or borax ($Na_2B_4O_7 \cdot 10\,H_2O$) as a potential sintering aid for $BaTiO_3$. Borax has multiple applications in ceramics, glass and enamels, as well as in nuclear shielding materials because of its neutron absorption properties [27]. To the best of our knowledge, borax has not been previously explored as a sintering aid for $BaTiO_3$. However, previous studies have demonstrated the effectiveness of borax in enhancing the sinterability and piezoelectric properties of other ceramics. For example, Chen *et al.* [28] studied the effect of borax addition on the sintering and electrical properties of $(K_{0.5}Na_{0.5})NbO_3$ (KNN) piezoceramics. Their results revealed that borax is an effective additive for improving the sinterability and thus the piezoelectric properties of KNN material. In another work, Kalemtas [29] utilized borax decahydrate as sintering aid in the α-Si_3N_4 material and CC31 commercial-grade kaolin system to fabricate a highly porous ceramics at low sintering temperature. He reported that the presence of high amount of borax results in formation of significant amount of liquid phase during the sintering process, and consequently lowers the sintering temperature of this ceramics.

The objective of this study was to produce the BT powder compacts to be sintered at lower temperatures by using a cheap additive material and simple procedure. This was achieved by using borax as a low melting temperature additive in small quantities. The effect of this addition on the physical and dielectric properties of $BaTiO_3$ ceramics was investigated in detail.

II. Experimental

$BaTiO_3$ (Alfa Aesar, Karlsruhe, Germany) was mixed with different amounts of borax (i.e. 1, 2, 5 and 10 wt.% of $Na_2B_4O_7 \cdot 10\,H_2O$) to prepare samples with different compositions denoted as BT1, BT2, BT5 and BT10, respectively. For each composition, borax was completely dissolved in distilled water and a binder mixture consisting of 1 wt.% polyvinyl alcohol and 0.5 wt.% polyethylene glycol was added. The mixture was stirred for 1 h, followed by the addition of $BaTiO_3$ powder. The slurry was dried at 90 °C overnight (NEY Furnace, M-525), crushed in an agate mortar (Fritsch, Germany) and sieved through a 112 µm mesh.

The powders were pressed into cylindrical pellets (12 mm in diameter) using a uniaxial hydraulic press at $3\,/cm^2$. The pellets were sintered at 1100 and 1150 °C for 3 h in a Carbolite furnace, GPC 1300, UK. For comparison, the pure BT ceramics without additive (denoted as BT0) was also prepared by sintering at 1350 °C.

Relative densities of the samples were determined using the Archimedes method. The phase compositions of the sintered samples were studied by X-ray, using XRD3A diffractometer Shimadzu, Japan. X'Pert High Score software program was used for the Rietveld refinement of the XRD diffraction data. Scanning electron microscope (SEM: Zeiss EVO10, Germany) was used to examine the microstructures of different samples. Prior to the electrical measurements, the different samples were polished using 320 grits, followed by thermal etching at 1050 °C/1 h. The pellets were then painted with silver paste on both sides, to make the two electrodes and finally fired at 650 °C for 30 min. Dielectric properties, including relative permittivity (ε_r) and dielectric loss ($\tan\delta$), were measured using an RCL meter (Philips PM6304) over a frequency range of 0.1–100 kHz.

III. Results and discussion

3.1. Structural characterization

The pellets containing small amount of borax (BT1 and BT2) exhibited good densification, after sintering at 1100 and 1150 °C, and reach relative densities above 90 %TD (Table 1). The densities were found to decrease with the increase of borax content, due to the low density of borax, and increase with the increase in the sintering temperature, but only slightly.

Table 1. Densities of BT1 and BT2 samples, sintered at 1100 and 1150 °C

Sample	Density [%TD]	
	1100 °C	1150 °C
BT1	92.8	93.3
BT2	91.8	91.9

The presence of the borax additive, having low melting temperature (743 °C), enables liquid phase sintering to start very early after binder removal, facilitating densification at lower temperatures. The sintering mechanism in this case comprises particle rearrangement, grain boundary diffusion through the liquid borax film and grains coalescence as a result of the liquid phase capillary force. These results agree with previously published data which showed that the presence of a liquid phase enhances the densification [23,24] and are comparable to those of $BaTiO_3$ compacts made from hydrothermally prepared fine powder and sintered at higher temperature [18]. They are also comparable to those obtained in another study after liquid phase sintering using boron oxide additive [22]. In contrast, the samples containing high borax concentrations (BT5 and BT10), showed significant deformation during sintering at same temperatures, likely due to excessive liquid phase formation and accordingly we excluded them from further analyses.

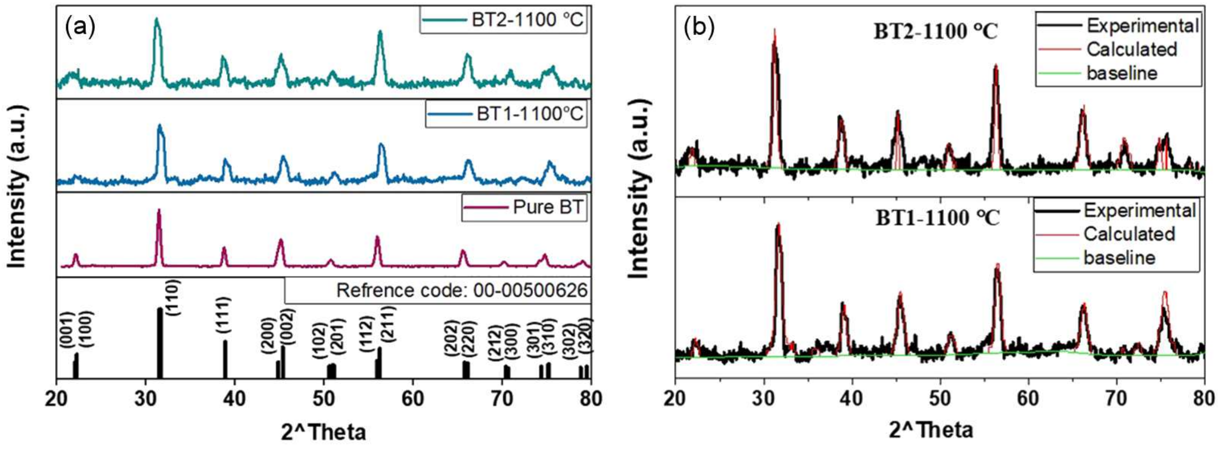

Figure 1. X-ray diffraction patterns of BT0 (pure BT, sintered at 1350 °C), BT1 and BT2 compacts sintered at 1100 °C/3 h: a) experimental results and b) Rietveld refinement results

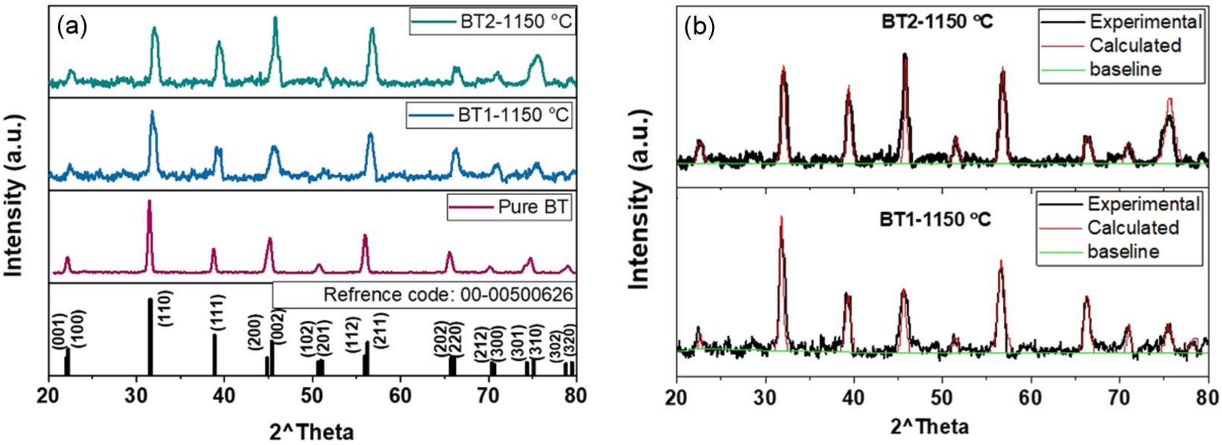

Figure 2. X-ray diffraction patterns of BT0 (pure BT, sintered at 1350 °C), BT1 and BT2 compacts sintered at 1150 °C/3 h: a) experimental results and b) Rietveld refinement results

Figures 1a and 2a show the XRD patterns of the BT0 (sintered at 1350 °C), BT1 and BT2 samples sintered at 1100 and 1150 °C, respectively. The patterns confirmed that all samples maintained the tetragonal perovskite structure of $BaTiO_3$, with no secondary phases observed, even for the samples sintered at 1100 °C. The slight splitting of (200) peak into (002) and (200) peaks observed for the samples sintered at 1150 °C, indicated tetragonal symmetry of $BaTiO_3$. The Rietveld refinement of the XRD data for the BT1 and BT2 samples (Figs. 1b, 2b) shows good agreement with the expected structure. It has been reported [30–32] that the tetragonality in barium titanate is closely related to the grain size and consequently to the sintering temperature.

SEM micrographs of the BT1 and BT2 samples sintered at 1100 and 1150 °C are shown in Figs. 3-5, and confirm that there are significant differences in microstructure of the sintered samples, influenced by both the borax content and the sintering temperature. The BT1 sample sintered at 1100 °C (Fig. 3a) exhibited a matrix of very small grains with some larger grains (up to 20 µm) distributed randomly in the matrix. The scattered powder agglomerates in the sample BT1 containing 1 wt.% of borax (effectively 0.5 wt.% of anhydrous borax) might have grown during sintering and produced this microstructure. The small amount of the borax added in this case might not have been enough to completely coat of barium titanate grains. On the other hand, one might not exclude the probability of heterogeneous distribution of the borax additive.

In contrast, a different microstructure is observed for the ceramics containing 2 wt.% of borax (BT2) and sintered at 1100 °C (Fig. 3b). The sample exhibited a bimodal grain size distribution, with both small (2–5 µm) and large (20–40 µm) grains surrounded by a liquid-phase grain boundary structure. The large grains contain domains and twins. Increasing the sintering temperature to 1150 °C promoted further grain growth and the microstructure of the BT1 sample (Fig. 4a) became more uniform, with a predominance of larger grains. More domains and twins are marked in Fig. 4b.

The BT2 sample sintered at 1150 °C (Fig. 5a) showed liquid phase grain boundary structure with predominate large grains of 40 µm in size. The increase in borax content increased the liquid phase which acted together with the increase in the sintering temperature, to pro-

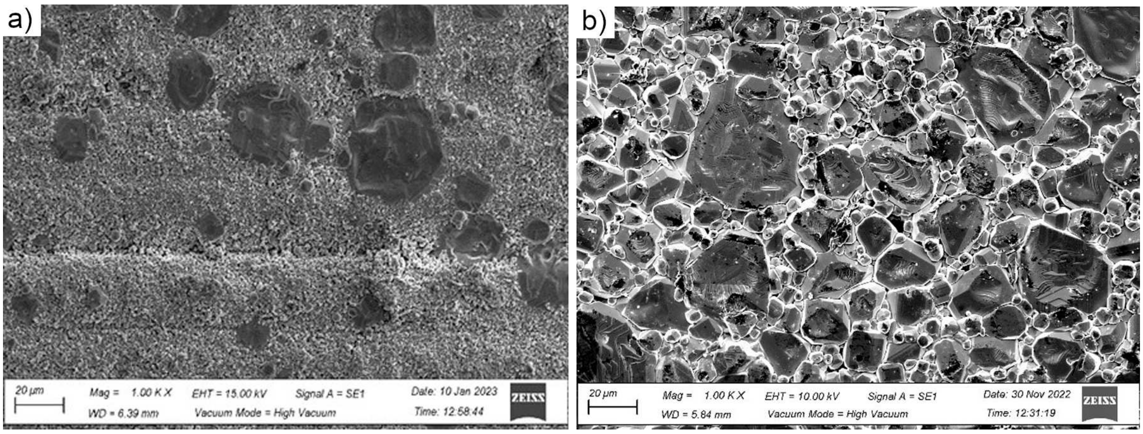

Figure 3. SEM micrographs of: a) BT1 and b) BT2 sample sintered at 1100 °C

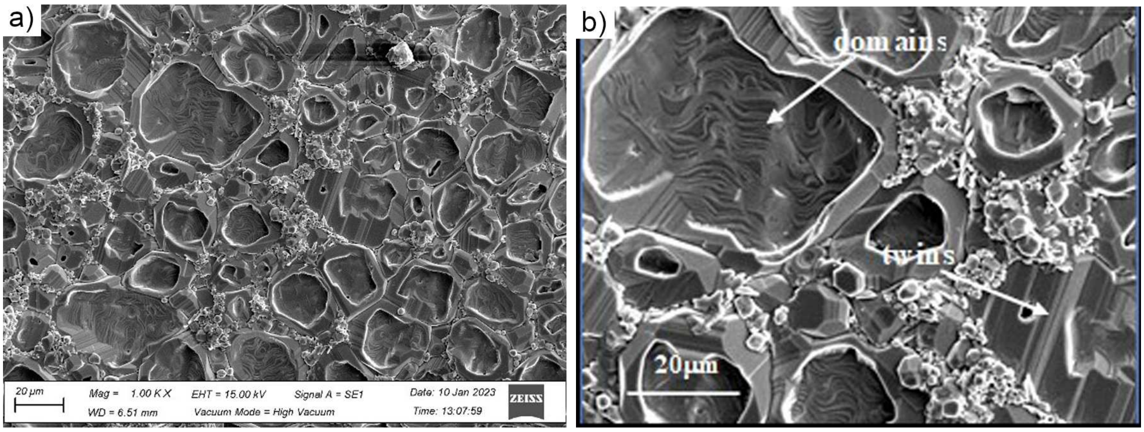

Figure 4. SEM micrographs of the BT1 sample, sintered at 1150 °C (a) and magnified area showing domains and twins (b)

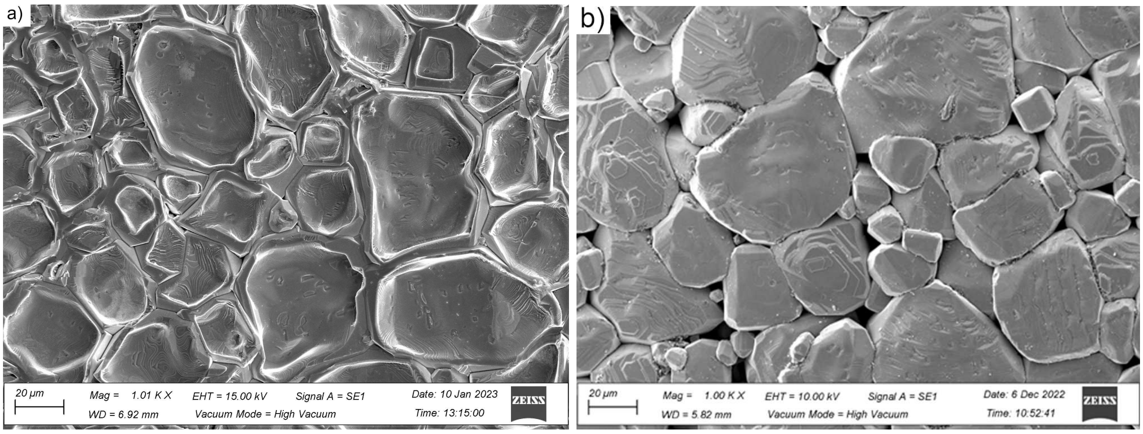

Figure 5. SEM micrographs of: a) BT2 sample sintered at 1150 °C and b) BT0 sample sintered at 1350 °C

mote grain growth. A similar microstructure, of large grains containing domains and twins, could be noticed for pure barium titanate (BT0) sintered at 1350 °C (Fig. 5b). This result agrees with previously published works [33,34] finding that the existence of both liquid phase and domains-twins promotes grain growth in barium titanate.

3.2. Dielectric properties

The dielectric properties of the sintered ceramics (Figs. 6-9) were measured as functions of temperature and frequency. The highest room temperature relative permittivity of 2410 was obtained for the BT1 sample sintered at 1100 °C (Fig. 6) with a noticeable decrease in the relative permittivity with frequency increase. However, its relative permittivity at the Curie temperature (T_C) is rather low (3080). These results reflected the grain size effect, where the major part of the BT1 sample sintered at 1100 °C consists of small grains. This trend is well known for the $BaTiO_3$ ceramics having a fine grained microstructure [23,35]. Fine-grained $BaTiO_3$ has a single domain inside each grain and the

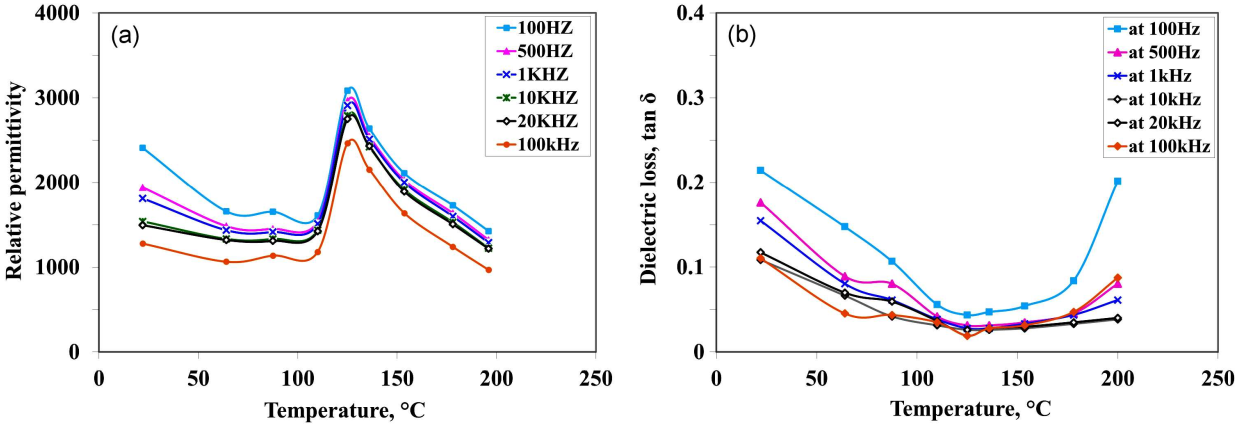

Figure 6. Relative permittivity (a) and dielectric loss (b) vs. temperature for the sample BT1 sintered at 1100 °C

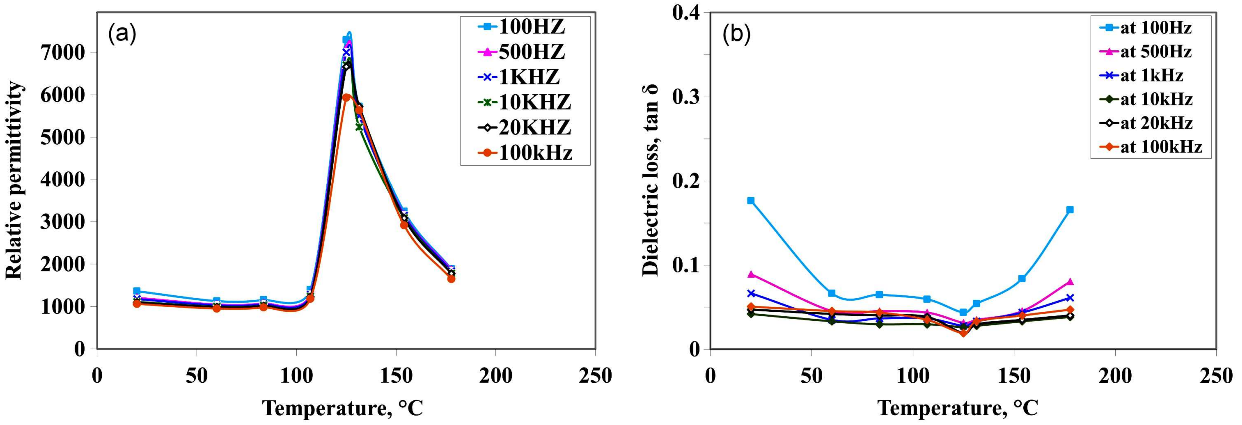

Figure 7. Relative permittivity (a) and dielectric loss (b) vs. temperature for the sample BT2 sintered at 1100 °C

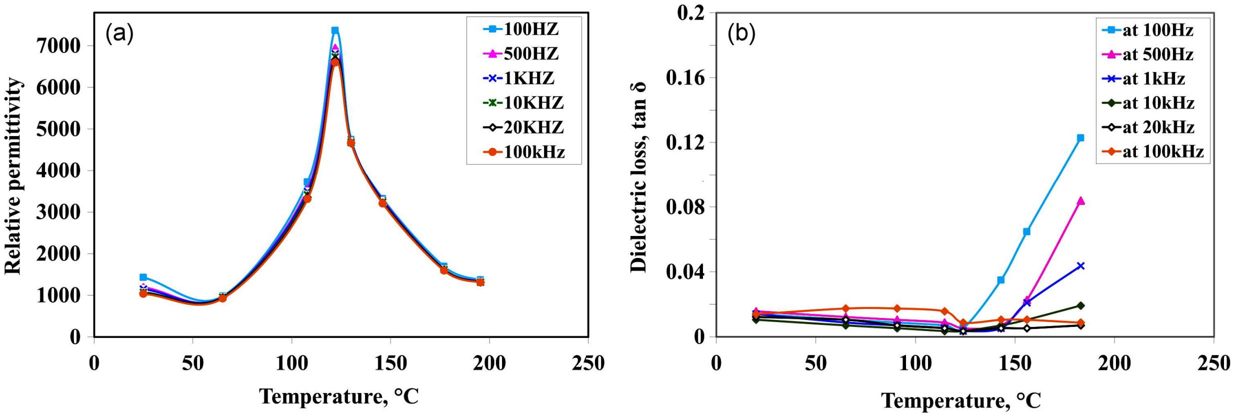

Figure 8. Relative permittivity (a) and dielectric loss (b) vs. temperature for the sample BT1 sintered at 1150 °C

grain boundaries limit the movement of domain walls, resulting in a low relative permittivity at the Curie point. Meanwhile, the internal stresses in fine grained $BaTiO_3$ are greater than in the coarse-grained material, which leads to a higher relative permittivity at room temperature [36].

Different behaviour concerning the frequency dependence of relative permittivity is observed for the sample BT2 sintered at 1100 °C (Fig. 7) and for both BT1 and BT2 samples sintered at 1150 °C (Figs. 8 and 9). The corresponding ε_r-T curves have a typical behaviour of the pure barium titanate (BT0) sintered at a higher temperature of 1350 °C (Fig. 10) and are characterized with higher relative permittivity values at the Curie temperature. The high relative permittivities resulted from the addition of borax which promoted sintering and grain growth. The large grains tend to have higher permittivity at the Curie point due to the reduced domain wall motion and lower internal stresses compared to fine-grained $BaTiO_3$ [35]. The dielectric loss values (tan δ) are very low for all samples, showing the lowest values for the sample BT2 sintered at 1150 °C.

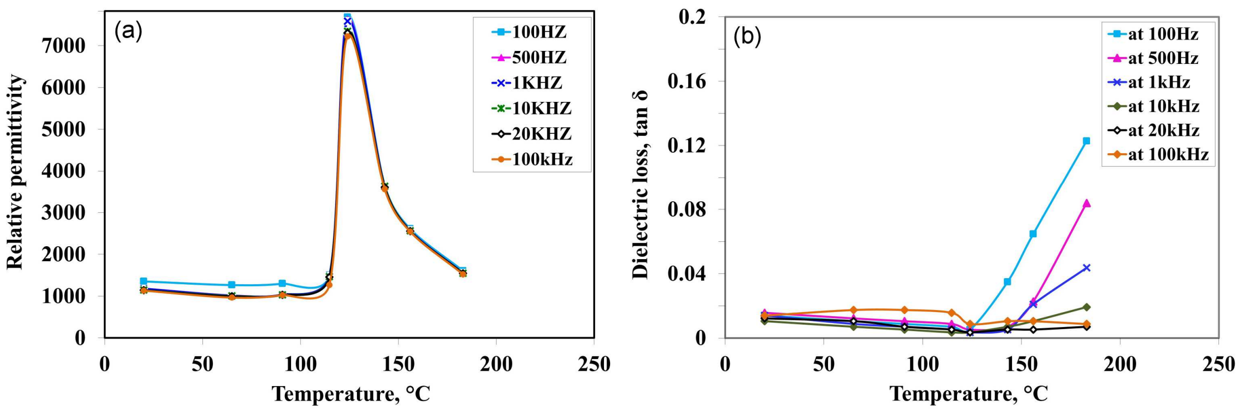

Figure 9. Relative permittivity (a) and dielectric loss (b) vs. temperature for the sample BT1 sintered at 1100 °C

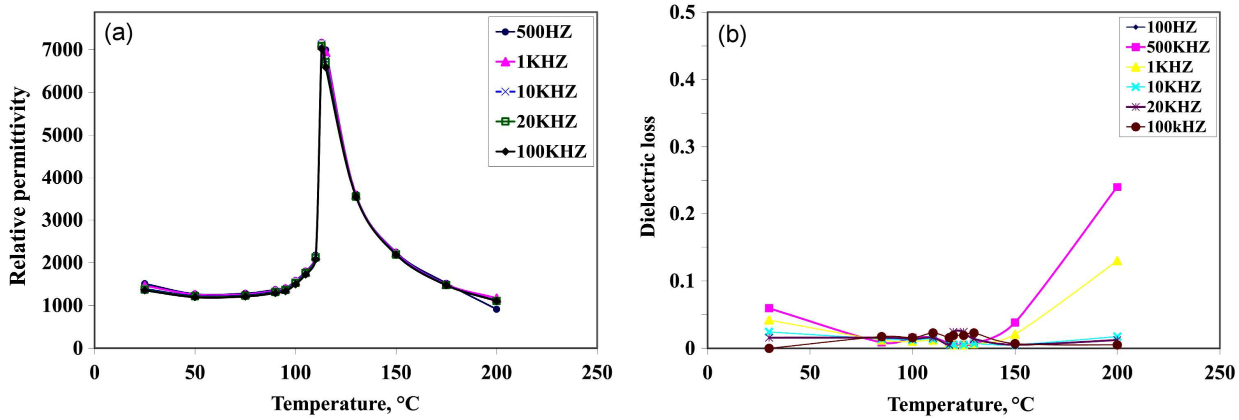

Figure 10. Relative permittivity (a) and dielectric loss (b) vs. temperature for the sample BT0 sintered at 1350 °C

For comparison, the relative permittivities of the BT1 and BT2 ceramics measured at room temperature (RT) and Curie temperature (T_C) and at frequencies 0.1 kHz and 1 kHz are given in Table 2. The present relative permittivity values obtained for the BT with borax additive and sintered at such low sintering temperatures are comparable to those for barium titanate without additive sintered at 1350 °C (Fig. 10), and are equivalent or even higher than those in recently published works on barium titanate sintered also at higher temperatures [11,31].

The permittivity changes significantly with temperature, i.e. it increases gradually with the rise in temperature up to its maximum value at the Curie temperature (T_C), then it decreases. At room temperature barium titanate, having ABO_3 perovskite structure and centro-asymmetric unit cell, exhibits spontaneous polarization. The polarization and consequently the relative permittivity increase in electric field with increasing temperature. At the Curie temperature, transition from ferroelectric state to paraelectric state takes place and the permittivity reaches its maximum. Above the Curie temperature, complete transformation from tetragonal to cubic phase takes place and movement of the Ti^{4+} ions becomes very limited i.e. there is no polarisation and consequently barium titanate becomes a perfect insulator having minimum dielectric loss. After this transformation to the centrosymmetric cubic phase, the relative permittivity decreases in accordance with Curie-Weiss law:

Table 2. The relative permittivities of BT1 and BT2 samples at 0.1 and 1 kHz

Sintering temperature	1100 °C		1150 °C	
	Frequency of 0.1 kHz			
	RT	T_C	RT	T_C
BT1	2410	3084	1472	6203
BT2	1365	7296	1353	7685
	Frequency of 1 kHz			
	RT	T_C	RT	T_C
BT1	1811	2790	1185	5663
BT2	1179.8	7000	1176	7588

$$\varepsilon = \varepsilon_0 + \frac{C}{T - T_C} \tag{1}$$

where, C is the Curie constant, T_C is the Curie temperature, ε_0 is the permittivity of the free space.

Figure 11 shows the temperature dependence of the reciprocal permittivity above the Curie temperature of the BT1 and BT2 samples sintered at 1100 and 1150 °C respectively. The linear trend above the Curie temperature can be observed in accordance with the Curie-Weiss law. The Curie constants for the samples ranged from

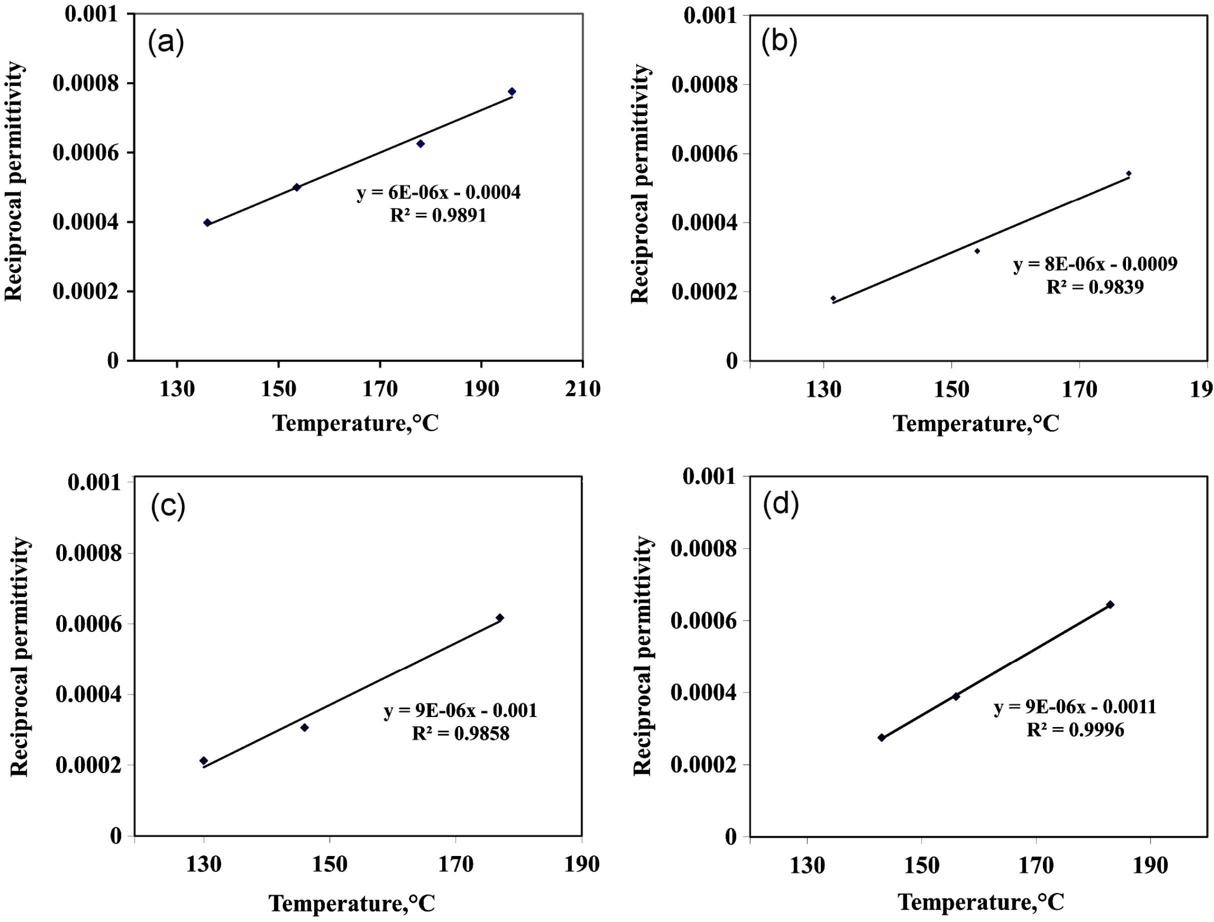

Figure 11. Reciprocal permittivity ($1/\varepsilon$) vs. temperature above the Curie temperature for samples: a) BT1 sintered at 1100 °C, b) BT2 sintered at 1100 °C, c) BT1 sintered at 1150 °C and d) BT2 sintered at 1150 °C

1.1×10^5 to 1.6×10^5 °C, which aligns with values reported for pure barium titanate by Babkair *et al.* [37], Paunović *et al.* [38] and Othman *et al.* [39].

IV. Conclusions

This study demonstrates that borax ($Na_2B_4O_7 \cdot 10\,H_2O$) is an effective sintering aid for $BaTiO_3$ ceramics, enabling densification at lower sintering temperatures. The key findings are:

- The presence of the liquid phase enhanced grain growth, accelerated the densification of the $BaTiO_3$ and lowered sintering temperature from ~1350 to 1100 °C.
- Sintered densities of more 90 %TD were obtained at temperatures as low as 1100 °C.
- The addition of small amounts of borax promotes densification without the formation of secondary phases or degradation of the dielectric properties.
- Typical barium titanate ε_r-T behaviour was obtained for the BT1 and BT2 ceramics (containing 1 and 2 wt.% of borax) sintered at 1100 and 1150 °C.
- The highest relative permittivity at room temperature (2410) was obtained for the fine-grained sample BT1 sintered at 1100 °C, while at the Curie temperature the highest relative permittivity (7685) was obtained for the coarse-grained sample BT2 sintered at 1150 °C.
- The $BaTiO_3$ ceramics having a higher amount of borax (2 wt.%) and sintered at 1100 or 1150 °C showed lower values of the dielectric loss ($\tan\delta$).
- The relative permittivity and relative density values obtained for the present $BaTiO_3$ materials sintered at such low sintering temperatures were higher than those for the pure $BaTiO_3$ sintered at higher temperatures.
- From the Curie-Weiss law the Curie constant ranged from 1.1×10^5 to 1.6×10^5 °C.

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