Microwave synthesis and sintering of \( \text{Ba}_{0.95}\text{Sr}_{0.05}\text{TiO}_3 \)

O.P. Thakur\textsuperscript{a}, Chandra Prakash\textsuperscript{a,*,} D.K. Agrawal\textsuperscript{b}

\textsuperscript{a}Solid State Physics Laboratory, Jakkur, Madras 600036, India  
\textsuperscript{b}Materials Research Laboratory, Pennsylvania State University, University Park, PA 16802, USA

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**Abstract**

Paper reports synthesis and structural properties of \( \text{Ba}_{0.95}\text{Sr}_{0.05}\text{TiO}_3 \) (BST) system. The material powder was prepared by solid state route and sintering was carried out using conventional furnace and microwave oven. XRD, SEM and thermal expansion studies were carried out for both the samples. It is found that the microwave sintered BST shows better densification than conventional furnace sintering, fine and uniform grain size, higher linear thermal expansion and improved dielectric properties. Possible mechanisms are explained.

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**Keywords:** Microwave; Synthesis; Sintering; BST; Ferroelectrics

**1. Introduction**

\( \text{Ba}_{0.95}\text{Sr}_{0.05}\text{TiO}_3 \) (BST) is a dielectric material with a perovskite structure that finds many applications in electronic devices. When pure, this material is highly resistive at room temperature but its electrical resistivity can be dramatically lowered by some dopants. The structural and electrical properties can be modified via partial substitution of either Ba-ions (A-site doping) or Ti ions (B-site doping). A-site doping with cations of the same valence as Ba causes the \( T_c \) (Curie temperature) to either decrease (Sr-substitution) or increase (Pb-substitution) without any significant broadening of the transition \cite{11}. The \( \text{BaTiO}_3 \)-based family of normal ferroelectrics is interesting candidates for field-induced piezoelectric transducers due to the large polarizations, large permittivities and large induced strains achievable. Conventional methods for the synthesis of ferroic phases require temperatures in the range of 900–1450 °C and several hours of soaking time. With the advent of microwave processing, it is possible to sinter the ferroic materials at astonishingly low time.

In the microwave process, the heat is generated internally within the material on account of absorption of microwave energy instead of originating from external sources, and hence there is an inverse heating profile. The heating is very rapid as the material is heated by energy conversion rather than by energy transfer, which occurs in conventional techniques. Microwave sintering has many advantages over conventional methods \cite{2–6}. Some of these advantages include time and energy saving, very rapid heating rates (>400 °C/min), considerably reduced processing time and temperature, fine microstructures and hence improved mechanical properties, it is environmentally

\textsuperscript{a} Corresponding author. Tel.: +91-11-3921602; fax: +91-11-3913609

E-mail address: cpramak@hotmail.com (C. Prakash)
friendly, and so on. Here, we report the synthesis of $\text{Ba}_{0.95}\text{Sr}_{0.05}\text{TiO}_3$ (BST) processed by microwave sintering method and also by conventional technique just to compare the properties. In this study, single phase $\text{Ba}_{0.95}\text{Sr}_{0.05}\text{TiO}_3$ (BST) was synthesized by microwave heating in much shorter time compared with conventional heating methods.

2. Experimental procedure

The synthesis material was started with powder mixture containing equimolar amount of AR grade, $\text{BaCO}_3$, $\text{SrCO}_3$, and $\text{TiO}_2$. The powder mixture was ball milled in distilled water and then calcined twice at 1050 and 1100 °C for 4 h. Calcined powders were isostatically pressed in a rod form at a pressure of 200 MPa using Cold isostatic press (M/s Autoclave Engineers). Cold isostatic process gives homogenous and better compaction of green rods. These rods were sintered at 1450 °C for 4 h in air atmosphere.

Microwave synthesis was conducted using a 1.5 kW, 2.45 GHz single mode microwave applicator. The heating rate was kept in a range of 100 °C/min (by controlling the input microwave power) for all synthesis procedures present. Density and microstructural information were obtained on microwave processed and conventionally processed samples by Archimedes principle and SEM, respectively. The quality and formation of the compounds were checked by X-ray diffraction technique. XRD patterns were recorded using Philips powder diffractometer with Cu-Kα radiation in a wide range of 2θ (20°≤2θ≤70°) at a scanning rate of 2°/min. Linear thermal expansion behaviour was studied using dilatometer (Orton 1600D) in the temperature range up to 250 °C.

3. Results and discussion

The XRD Patterns of the conventionally and microwave synthesized $\text{Ba}_{0.95}\text{Sr}_{0.05}\text{TiO}_3$ (BST) powders are shown in Fig. 1. All these patterns depict the formation of single phase with tetragonal structure. The lattice parameters of the ceramic samples were estimated from refined parameters calculations from peaks in an interval of 20 – 70°.20 and shown in Table 1. The diffractograms reveal a unique tetragonal phase for both the samples within the resolution of the experiments. The lattice parameter $a$ and $c$ increased slightly for microwave sintered BST sample while tetragonality ($c/a$) is slightly diminished that is due to the enhanced diffusion of Sr-ion into the perovskite lattice as microwave sintering process results in enhanced sintering. This is further confirmed from increased volume of lattice in the case of microwave sintering process.

Comparison of the time–temperature profiles for BST Synthesis in Fig. 2 indicates unambiguously the energy and time efficiency of microwave processing over conventional method. The microstructures of the fractured surfaces of conventionally and microwave processed BST samples are shown in Fig. 3. The increase in grain size (~18 μm) is not fully understood and needs further investigation but could be due to a phase inducing a narrow range of greater solubility and excess liquid phase that is clearly visible in the micrograph (Fig. 3a). The grain size is fairly uniform for all the samples and ranges between 1 and 20 μm and the grain size observed in microwave processed specimens are smaller and the sample is denser than conventional one. The rapidity of microwave method also avoids undesirable grain growth and provides a finer and uniform microstructure (Fig. 3), which is an attractive feature for the processing of electroceramics [7]. This is also clearly indicated from thermal expansion behaviour of these two samples as...
Table 1
Structural and dielectric parameters for conventionally and microwave sintered Ba_{0.6}Sn_{0.4}TiO_3 ceramics

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Conventionally sintered</th>
<th>Microwave sintered</th>
</tr>
</thead>
<tbody>
<tr>
<td>ε (Å)</td>
<td>4.0276</td>
<td>4.0276</td>
</tr>
<tr>
<td>μ (Å)</td>
<td>3.9804</td>
<td>3.9986</td>
</tr>
<tr>
<td>εμ</td>
<td>1.0093</td>
<td>1.0073</td>
</tr>
<tr>
<td>Volume (Å³)</td>
<td>63.6537</td>
<td>64.3982</td>
</tr>
<tr>
<td>% Porosity</td>
<td>6</td>
<td>3</td>
</tr>
<tr>
<td>Dielectric constant (ε')</td>
<td>1350</td>
<td>1450</td>
</tr>
<tr>
<td>Dissipation factor (tanδ)</td>
<td>0.0349</td>
<td>0.0217</td>
</tr>
<tr>
<td>Thermal expansion coefficient (α in °C)</td>
<td>0.0014</td>
<td>0.00129</td>
</tr>
</tbody>
</table>

shown in Fig. 4 which indicates the magnitude of thermal expansion is more for microwave processed sample. However, the thermal expansion coefficient (α) (listed in Table 1) in paraelectric region is more for conventionally sintered sample. The transition temperature (T_c) is found to be little higher in microwave sintered sample which can be ascribed, as it is reported, to domain arrangements in microwave sintered specimens being more complicated, than those in conventional one [8]. The formation of complicated domain arrangements clearly implies that a large strain field is induced when the sample cools from the paraelectric region. This might be explained by the interaction of microwave and the atomic species as well as the interaction of thermal stress and local lattice distortion in the materials. The domain mobility

Fig. 2. Comparison of the time-temperature profiles for microwave and conventional synthesis of Ba_{0.6}Sn_{0.4}TiO_3 ceramics.

![Fig. 3. Scanning electron micrographs for (a) conventional and (b) microwave sintered Ba_{0.6}Sn_{0.4}TiO_3 ceramics.]

![Fig. 4. Thermal expansion behaviour of Ba_{0.6}Sn_{0.4}TiO_3 ceramics.]
is restricted in the specimens due to lattice distortions, which influence the Curie temperature of microwave sintered specimen. Also the increase of Curie temperature may be ascribed to the increase of lattice constant in the case of microwave sintered sample [9]. Interestingly, our preliminary dielectric measurements indicate that the dielectric constant of the microwave sintered BST ceramic is higher than that for conventionally sintered BST sample. The dielectric parameters are included in Table 1. Detailed electrical properties are being published separately.

4. Conclusions

The use of microwave energy resulted in the formation of single phase BST with tetragonal structure in just 20 min. Microwave sintered samples show improvement in dielectric loss tangent. Microwave sintering of BST leads to higher densification in much shorter time duration and finer microstructure compared to conventional procedures. The microwave method is found to be simple, fast and quite general for the preparation of technologically important electroceramics.

References