



Preparation and dielectric properties of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ layered Perovskite at microwave frequencies

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Abstract

The layered $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ perovskite ceramic was synthesized via conventional solid state mixed oxide sintering process. The material was sintered in a range of 1375 °C to 1500 °C for 4 h to develop dense ceramic. Relative density of 97% was achieved while sintering at 1450 °C for 4 h. The XRD data analysis confirmed single phase layered perovskite and the microstructure comprised well-connected plate-like grains with an average grain size of $\sim 5.46 \mu\text{m}$ as determined by ImageJ software. The material offers a good combination of microwave dielectric properties such as $\epsilon_r \sim 57$, $Q_{uf} \sim 9500 \text{ GHz}$ and $\tau_f \sim 30 \text{ ppm/}^\circ\text{C}$.

Keywords: microwave dielectric ceramics, layered perovskite, $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$, dielectric resonator.

1 Introduction

Dielectric resonator is an important electronic component used in telecommunication systems that function when resonating at a particular frequency called resonant frequency. Such materials are used in mobile phone base stations as filters and resonators in cell phone [1–3]. The high dielectric constant values can enable the miniaturization of microwave devices as the size of resonator is linked to ϵ_r via the relation $\epsilon_r \sim \frac{1}{\sqrt{D}}$, when the resonant frequency of microwave is kept constant [1, 4, 5]. Dielectric ceramics having $\epsilon_r > 20$, a high Q-factor ($Q > 2000$) at the resonant frequency (f_0), and a τ_f value near 0 ppm/°C are required for microwave dielectric resonator applications [6, 7]. Several dielectric materials have got their practical applications as dielectric resonators and filters in mobile handsets and mobile base stations [8–10]. However, a continuous search is underway for dielectric materials with ultra-low losses, high ϵ_r and a near-zero τ_f .

$\text{A}_5\text{B}_5\text{O}_{17}$ layered perovskites have attracted considerable attention due to their good microwave dielectric characteristics [11–15]. $\text{SrO-Nb}_2\text{O}_5\text{-TiO}_2$



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ternary system was investigated by several researchers [14–16]. $\text{Sr}_5\text{Nb}_4\text{TiO}_{17}$ possesses orthorhombic crystal structure and offer high $\varepsilon_r = 74$, $Q_{\text{ufo}} = 1303 \text{ GHz}$ but high positive $\tau_f = +332 \text{ ppm}/^\circ\text{C}$ [17] while its Ta analog ($\text{Sr}_5\text{Ta}_4\text{TiO}_{17}$) offer $\varepsilon_r = 66$, $Q_{\text{ufo}} = 8500 \text{ GHz}$ and $\tau_f = 180 \text{ ppm}/^\circ\text{C}$ [18]. $\text{Ca}_5\text{Nb}_4\text{TiO}_{17}$ has been reported to offer moderate $\varepsilon_r = 44.9$, high $Q_{\text{ufo}} = 17,600 \text{ GHz}$ and $\tau_f = -112 \text{ ppm}/^\circ\text{C}$ [19] while its Ta analog ($\text{Ca}_5\text{Ta}_4\text{TiO}_{17}$) offer $\varepsilon_r = 40.1$, $Q_{\text{ufo}} = 16,450 \text{ GHz}$ and $\tau_f = -53.6 \text{ ppm}/^\circ\text{C}$ [19]. As it is evident, that in general Sr containing layered materials have +ve value of τ_f while Ca based layered materials have -ve value of τ_f . The reason is that Sr has higher polarizability and creates more “rattling space” or in other words provides a less constrained environment for the oxygen octahedra. Upon increasing the temperature, the change in the polarizability of the lattice and the structural expansion actually works in a way that increases the dielectric constant significantly ($\tau_\varepsilon > 0$), leading to a positive τ_f . Conversely, Ca is smaller in size than Sr and fits more “tightly” within the octahedral framework, creating a more rigid and compact structure. In this case, the thermal expansion dominates the dielectric behavior differently, leading to a decrease in ε_r as temperature increases ($\tau_\varepsilon < 0$), resulting in a negative τ_f . Ba, having a larger ionic radius and a higher dielectric polarizability than Ca, suggests that partial replacement of Ca by Ba in $\text{Ca}_5\text{Nb}_4\text{TiO}_{17}$ may tune its τ_f value toward zero while increasing the ε_r value.

Therefore, in the present study $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ was prepared and the microwave dielectric properties were investigated to obtain temperature stable microwave ceramics.

2 Experimental Procedure

$\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic was fabricated through conventional solid state sintering method. BaCO_3 (~99.9%), CaCO_3 (~99.99%), Nb_2O_5 (~99.95%) and TiO_2 (99.9% rutile) were weighed in stoichiometric ratios via a digital electronic balance followed by milling using ball milling process for 24 h in ethanol. The slurry was kept at $\sim 95^\circ\text{C}$ in a drying oven followed by heating at 1300°C for 5 h at a heating and cooling rate of $3^\circ\text{C}/\text{min}$. The resulting calcined powders were milled again to obtain fine powders with small particles.

10 mm diameter and 7 mm thick green body pellets were made from the calcined powders pressed at

100 MPa followed by heating at high temperature for sintering at different temperatures in the range of 1375°C to 1500°C for 4 h at a heating rate of $5^\circ\text{C}/\text{min}$.

The faces of optimally sintered samples were polished to remove the impurity that developed during sintering process on the surfaces. The bulk densities were determined via the Archimedes principle to determine the optimized sintering temperature. The optimally dense sintered pellets were crushed to make fine powders followed by phase analysis via powdered XRD technique. The microstructural examination was carried out for polished and thermally etched surfaces using scanning electron microscope. The microwave dielectric properties were determined using a vector network analyzer (E8363B, Agilent, United States) using cavity resonator technique. Resonant frequencies at two different temperatures i.e at 20°C and 80°C were measured and denoted at f_{20} and f_{80} . The τ_f values measured in the unit of $\text{ppm}/^\circ\text{C}$ were determined using Eq (1).

$$\tau_f = \frac{f_{80} - f_{20}}{f_{20}(T_{80} - T_{20})} \times 10^6 \quad (1)$$

where T_{20} and T_{80} are 20°C and 80°C , respectively.

3 Results and Discussion

The determination of relative density (ρ_{rel}) is very important as it affects the final performance of a material. The relative density (%) was calculated by dividing the measured bulk density by the theoretical density and multiplying by 100.

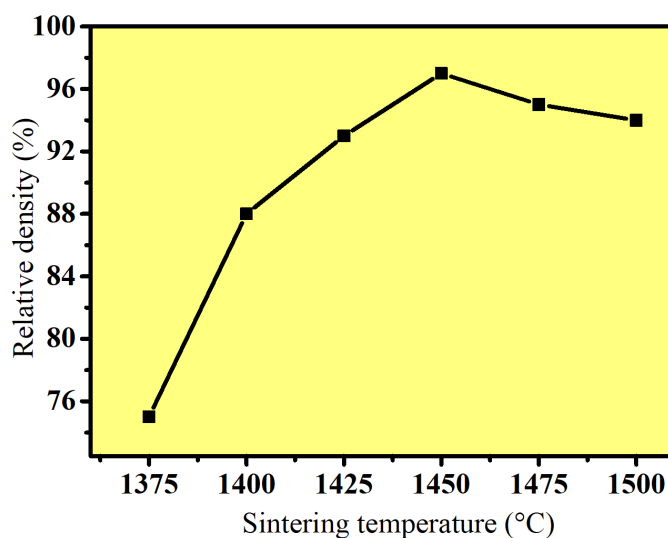


Figure 1. The relative density (ρ_r) of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic as function of sintering temperature.

The relative density of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic was plotted as a function of sintering temperature as depicted in Figure 1. It was observed that the relative density monotonically increased with increasing sintering temperature from 1375°C , reaching a maximum value of $\sim 97\%$ at 1450°C , and then decreased to $\sim 95\%$ with a further increase in sintering temperature to 1500°C . The initial monotonic increase in density could be the result of uniform grain growth where the ionic diffusion is uniform with sintering temperature from 1375°C to 1450°C . The final decrease with increasing sintering temperature above 1450°C could be linked to the non-uniform grain growth leading to the formation of pores [18].

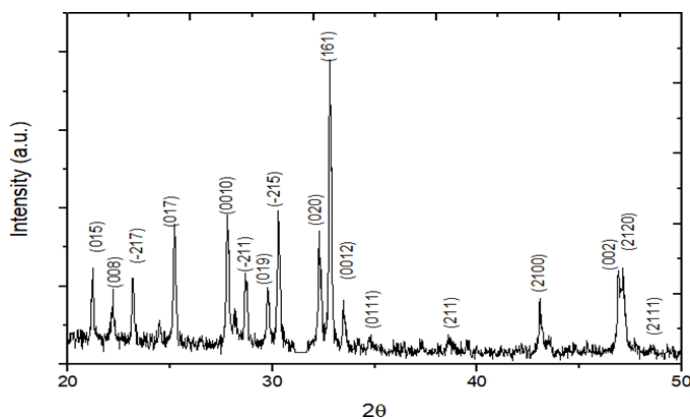


Figure 2. X-ray diffraction pattern from powdered sample of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic sintered at 1450°C .

The X-ray diffraction pattern obtained from the powdered $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic sintered at 1450°C for 4 h is displayed in Figure 2. The spectrum contains the intensities of diffracted X-rays photons as a function of diffraction angle (2θ) in the range of 20 to 50 degrees,

where the major peaks lie. The peaks were identified using software, and the corresponding intensities and d -values matched those of a standard pattern of $\text{Ca}_5\text{Nb}_4\text{TiO}_{17}$ ceramic. It is observed that a single phase has been detected within the XRD detection limit with orthorhombic crystal structure. A single phase formation is beneficial for achieving good microwave dielectric properties for this ceramic.

The lattice parameters a , b and c were calculated using least square method and are $5.60 \text{ \AA}$, $32.30 \text{ \AA}$ and $3.90 \text{ \AA}$ respectively. The theoretical density was calculated using Eq (2).

$$\rho_{th} = \frac{Z \times M}{N_A \times V_{unit}} \quad (2)$$

where Z is the number of formula unit per unit cell ($Z = 2$ in this case), M is the molecular weight (989.11 g/mol for $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$), N_A is the Avogadro number (6.022×10^{23}) and V_{unit} is the volume of the unit cell ($705.432 \text{ \AA}^3$). The calculated theoretical density was found to be $\sim 4.66 \text{ g/cm}^3$.

The grains distribution and their sizes contribute to the performance of material at microwave frequencies. The scanning electron microscope image obtained from thermally etched $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic sintered at 1450°C for 4 h via the secondary electron detector is shown in Figure 3(a). The microstructure is comprised of plate like grains which is typical morphology of layered perovskite ceramics. The average grain size was $5.46 \mu\text{m}$ as calculated using ImageJ software and the grain size distribution histogram is shown in Figure 3(b). This compact microstructure will have

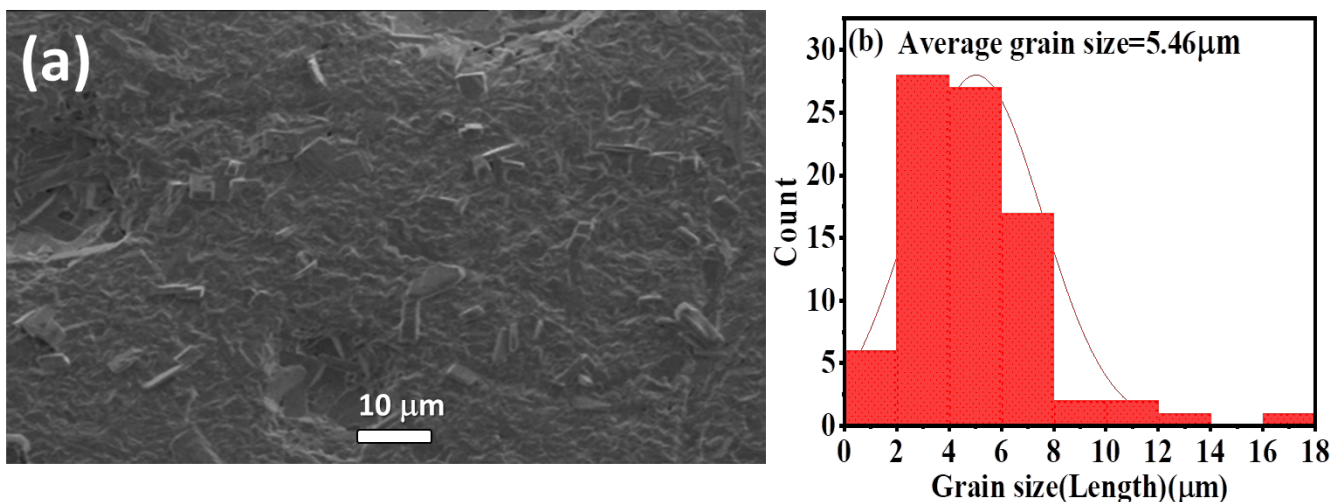


Figure 3. The scanning electron microscope image of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic sintered at 1450°C for 4 h.

impact on the microwave dielectric properties. Dense microstructure with almost no porosity is observed for this material composition.

The dielectric constant (ϵ_r) of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic at each sintering temperature was measured using the relation given by Eq (3) and its variation with sintering temperature is shown in Figure 4 [1].

$$\epsilon_r = \sqrt{\frac{C}{Df_o}} \quad (3)$$

where C , D and f_o are the speed of light, diameter of the sample under test and resonant frequency respectively. The variation of the dielectric constant followed the same trend as the relative density with sintering temperature. A previous study reported $\epsilon_r \sim 47.2$ for the Sr counterpart ($\text{SrCa}_4\text{Nb}_4\text{TiO}_{17}$) sintered to approximately 95% relative density [20]. The Ba^{2+} ion has been reported to have a higher dielectric polarizability of $6.40 \text{ \AA}^3$ than Sr^{2+} ($4.24 \text{ \AA}^3$) [21]. Therefore, the large ionic dielectric polarizability of Ba can cause higher ϵ_r of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ than its Sr counterpart ($\text{SrCa}_4\text{Nb}_4\text{TiO}_{17}$). Therefore, in the present study, a maximum $\epsilon_r \sim 57$ is achieved for $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ when sintered the ceramic pellet at 1450°C for 4 h as shown in Figure 4.

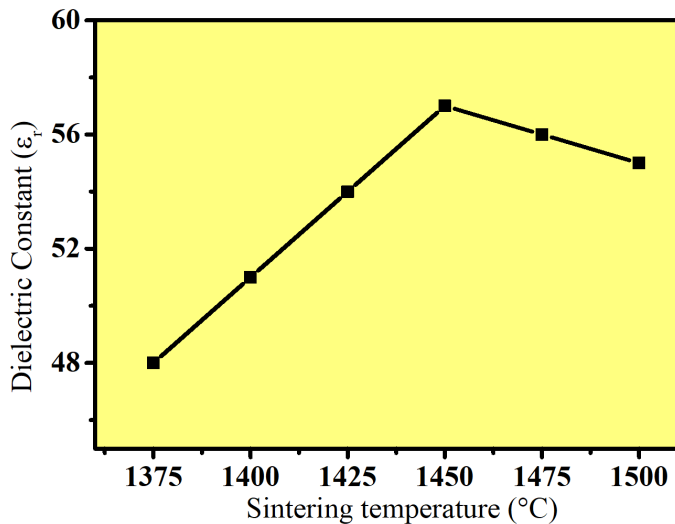


Figure 4. The ϵ_r of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramics as a function of sintering temperature.

Another very important characteristic of a dielectric resonator is the dielectric loss or its inverse the unloaded quality factor (Q_u). Generally in microwave frequencies, the product of Q_u and resonant frequency (f_o) are considered for comparison at different sintering temperatures. Both intrinsic and extrinsic

factors are taken into consideration when evaluating the $Q_u f_o$ value. The phonon contribution due to vibration of atoms bounded together is actually the intrinsic factor. The porosity, impurities, humidity and other crystal defects are termed as the extrinsic factors. In this study, single phase with almost no porosity is obtained and extrinsic contributions to the dielectric loss or the quality factor are considerably minimized. The Q_u is calculated using Eq (4) [1].

$$Q_u = \frac{f_o}{\Delta f} \quad (4)$$

where f_o is the resonant frequency and Δf is the bandwidth. This equation demonstrates that lower is the bandwidth; higher will be the Q_u value. The $Q_u f_o$ value also follows the trend as the density did with the sintering temperature as shown in Figure 5. The materials delivered its maximum $Q_u f_o \sim 9500 \text{ GHz}$ when sintered at 1450°C for 4 h.

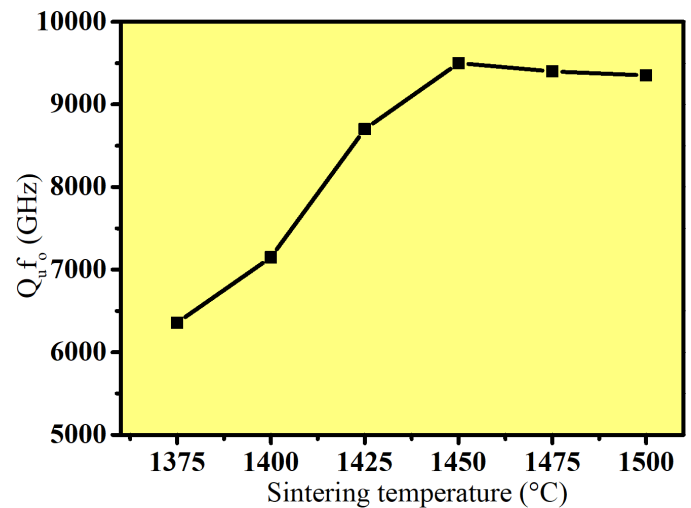


Figure 5. The $Q_u f_o$ of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic as a function of sintering temperature.

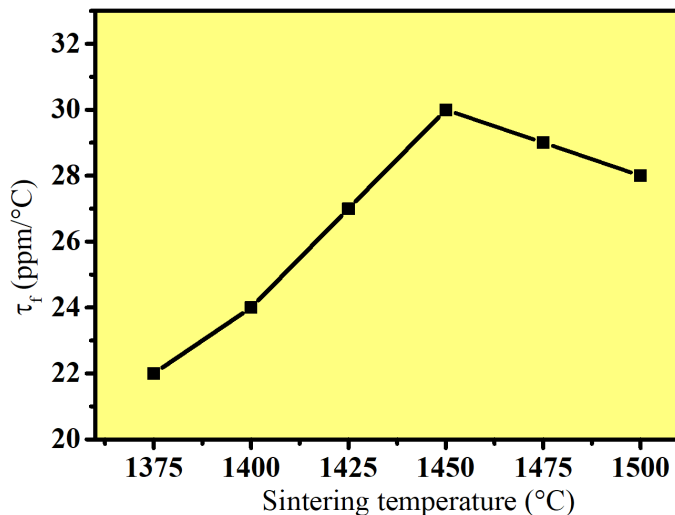
The third important characteristic is the temperature coefficient of resonant frequency (τ_f) to be considered for microwave material. The secondary phase, porosity and other defects also contribute to the overall τ_f value of a material.

In the present study, the τ_f of $\text{BaCa}_4\text{Nb}_4\text{TiO}_{17}$ ceramic is obtained to be $30.5 \text{ ppm}/^\circ\text{C}$ at a sintering temperature of 1450°C as shown in Figure 6.

The microwave dielectric properties of the present material are compared with the layered materials previously reported in the literature as given in Table 1. The current material exhibits a relatively high ϵ_r value and a significantly reduced positive τ_f compared with other materials in the same family.

Table 1. Comparison of the microwave dielectric properties of BaCa₄Nb₄TiO₁₇ ceramic with the same layered perovskites reported in the literature.

Compositions	ε_r	Q_{uf} (GHz)	τ_f (ppm/°C)	Ref
Sr ₅ Nb ₄ TiO ₁₇	74	1303	332	[17]
Ca ₅ Nb ₄ TiO ₁₇	44.9	17600	−112	[19]
Ca ₅ Ta ₄ TiO ₁₇	40.1	16450	−53.6	[19]
SrCa ₄ Nb ₄ TiO ₁₇	47.2	11984	−78.6	[20]
NaCa ₃ SrNb ₅ O ₁₇	57	4628	−41	[22]
BaCa ₄ Nb ₄ TiO ₁₇	57	9500	30.5	This work

**Figure 6.** The τ_f of BaCa₄Nb₄TiO₁₇ ceramic as a function of sintering temperature.

BaCa₄Nb₄TiO₁₇ has $\varepsilon_r \sim 57$ which is greater than that of Ca₅Nb₄TiO₁₇, due to the fact that the total dielectric polarizability of BaCa₄Nb₄TiO₁₇ is larger than Ca₅Nb₄TiO₁₇. On the other hand, BaCa₄Nb₄TiO₁₇ has lower ε_r than Sr₅Nb₄TiO₁₇ due to the fact that total dielectric polarizability of BaCa₄Nb₄TiO₁₇ is lower than Sr₅Nb₄TiO₁₇.

4 Conclusion

Microwave dielectric materials play a very important role in the field of telecommunication. The dielectric constant, quality factor (the inverse of dielectric loss), and temperature coefficient of resonant frequency are the three important properties to be determined at microwave frequencies for dielectric resonator applications. In this study, the phase, microstructure, and microwave dielectric properties were determined using XRD, electron microscopy, and a cavity resonator technique. XRD data analysis confirmed single phase BaCa₄Nb₄TiO₁₇ layered perovskite. Microstructural analysis demonstrates densely packed grains with a high relative density of 97% of the theoretical density

after sintering at 1450 °C for 4 h. Good combination of microwave dielectric properties such as $\varepsilon_r \sim 57$, $Q_{uf} \sim 9500$ GHz and $\tau_f \sim 30.5$ ppm/°C are obtained for the investigated ceramic.

Data Availability Statement

Data will be made available on request.

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Conflicts of Interest

The authors declare no conflicts of interest.

AI Use Statement

The authors declare that no generative AI was used in the preparation of this manuscript.

Ethical Approval and Consent to Participate

Not applicable.

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