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Sintering Processes and Dielectric Properties of Ceramic Material ($Ba_{0.4}Ca_{0.6}$) Ti_4O_9

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Abstract

Dielectric properties of ($Ba_{0.4}Ca_{0.6}$) Ti_4O_9 ceramic material prepared by mixed oxide solid state route were studied. X-Ray Diffraction (XRD) showed that the calcined powder of ($Ba_{0.4}Ca_{0.6}$) Ti_4O_9 at 900°C crystalizes in the phase of complex peroveskite type. Dielectric properties measurement was carried out by LCR meter at the range of (1 MHz to 100 MHz) and (1 GHz to 2 GHz) frequencies. The εr value of ($Ba_{0.4}Ca_{0.6}$)Ti_4O_9 sintered ceramic sample increased with increasing frequency. In the present work, a controlled mixed oxide solid state processing route was used to prepared microwave dielectric material ($Ba_{0.4}Ca_{0.6}$) Ti_4O_9 and microwave dielectric properties were measured at relatively lower and higher frequencies.

Keywords: Solid state route • Dielectric properties • Barium tetra titanate

About The Study

Due to its excellent ferroelectric and piezoelectric properties, to which is added the very best stability of its mechanical and chemical peroveskite structure, Barium Tetra Titanate (BT_4) remains the preferred material for many applications i.e. dielectric capacitors, reported by Hung, et al., [1] ceramics with Positive Temperature Coefficient of Resistance (PTCR) [2]. $BaTi4O_9$ (BT_4) is a dielectric material used in the microwave field [3]. BT_4 was first reported by Rase and Roy [4] in their study of the BaO-TiO_2 system. BT_4 was investigated as microwave material by O'Bryan, et al., [5-7]. The dielectric properties of BT_4 with several different additives have also been investigated in the microwave region [8-11]. The addition of WO_3 to the system BaO-TiO_2 results in multiple phases including $BaTi_4O_9$, $Ba_2Ti_9O_{20}$, $BaWO_4$, and TiO_2 [9]. The BaO-$4TiO_{2-0.1}WO_3$ ceramic was found to possess excellent microwave properties as dielectric constant i.e ϵr=35. Other important microwave ceramic material is based on the BaO-Nd_2O_3-TiO_2 system. Kolar, et al., [12] and Negas, et al. [13] determinate phase diagram and investigated the dielectric properties at 1 MHz in this system.

Several doping elements can be added to BT_4 ceramics for achieving some better properties for certain applications, Pb^{2+} would increase its transition temperature Tc, Sr^{2+} would decrease TC and Co^{2+} would attenuate the losses for intense electric field without affecting its piezoelectric constant [14]. Veenhuis et al., reported that BT_4 crystals show promising applications in the field of electronic or optical storage devices, advance laser technologies etc. [15]. Excellent efforts have been devoted to elucidate the effect of calcium doping on the dielectric properties of ($Ba_{0.4}Ca_{0.6}$) Ti_4O_9 solid solution.

In fact, calcium acts as a reduction inhibitor in BT_4 and decreases the possibility of formation of the unwanted hexagonal phase [16]. In the present work, a controlled mixed oxide solid state processing route was used to prepared microwave dielectric material ($Ba_{0.4}Ca_{0.6}$) Ti_4O_9 and microwave dielectric properties were measured at relatively lower and higher frequencies.

Experimental procedure

$CaCO_3$, TiO_2 and $BaCO_3$ were selected as reactants raw materials to prepare ($Ba_{0.4}Ca_{0.6}$) Ti_4O_9 ceramic material for microwave dielectric properties. High purity starting materials were weighted accordingly to stoichiometric ratios. The mixture of raw powders were ground in distilled water for 24 h in a horizontal ball mill with zirconia balls of 5 mm as a grinding media. The prepared powders were dried in oven at 90°C for 24 h in air. The dried mixture was calcined at 900°C for 4 h in air at 10°C/min heating/cooling rate. The calcined reagent was grinded manually with a pistol and mortar to avoid agglomeration.

The fine powder was pressed into pellets of 10 mm in diameter and 5 mm thick under a pressure of 100 MPa. The pellets samples were sintered at temperatures of 1300°C for 2 hrs in air with heating/cooling rates 10°C/min. The crystalline phases of the calcined ($Ba_{0.4}Ca_{0.6}$)Ti_4O_9 ceramic material sample was identified by using X-rays Diffractometer (XRD) (JDX-3532, JEOL, Japan) with Cu Kα (λ=0.15406 nm) radiation operated at 40 mA and 40 kV. The dielectric property of sintered ceramic sample was measured at microwave frequencies by LCR meter (Agilent 4287A).

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Figure 1 shows the XRD pattern of the $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ calcinedceramics at 900°C for 4 h in air. The XRD pattern shows the hexagonal structure of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sample with density 4.085 gm/cm^3 and volume 0.148 cm^3. When Ba^{2+} replaced by Ca^{2+}, the $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ ceramics would form solid solution. The phase transition occurs from orthorhombic structure of BT_4 peroveskite to hexagonal of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ by doping Ca^{2+} contents. Table 1 shows the microwave dielectric properties of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sintered ceramics at 1300°C for 2 h. The variations of εr and Q × f were consistent with the frequency. Maximum εr (38.66), Q × f (23059 MHz) and low dielectric loss (0.0043) values were observed at 100 MHz frequency as shown in Figures 2 and 3. At higher frequency (1.8 GHz) the dielectric constant value found to be 67.88 as shown in Figure 4. The εr value of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sintered ceramic sample increased with increasing frequency. Figure 5 shows the Q × f and tan (σ) at the range of frequency 1 GHz to 2 GHz.

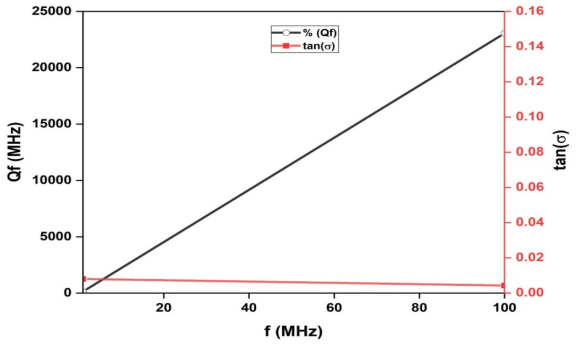

Figure 2. Dielectric constant (εr) of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sintered ceramics at 1300°C. **Note:** (—○—)%(Qf), (—■—) tan(σ)

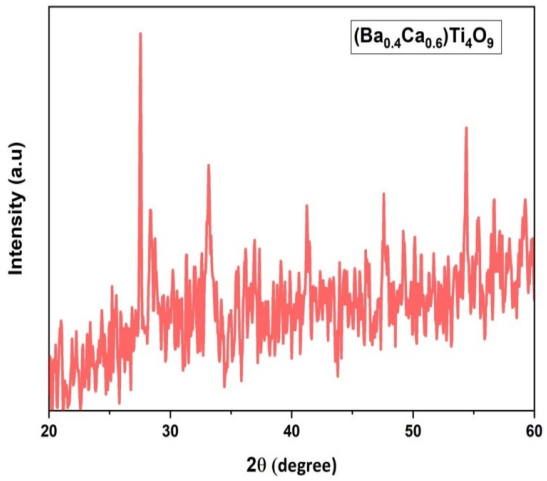

Figure 1. XRD pattern of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ calcined ceramics at 900°C for 4 h.

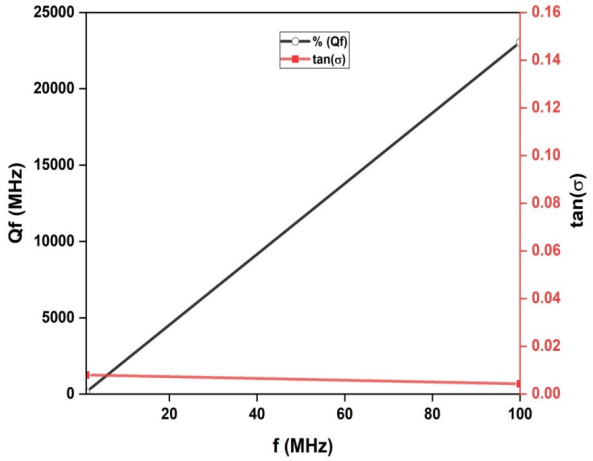

Figure 3. Qf and tan (δ) of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sintered ceramics at 1300°C. **Note:** (—○—)%(Qf), (—■—) tan(σ)

Table 1. Processing conditions and Microwave dielectric properties of $(Ba_{0.4}Ca_{0.6})Ti_{4O9}$ sintered ceramics at 1300°C.

C.T (°C)	S.T (°C)	f	εr	Qf	tan (σ)
900°C/4 h	1300°C/2 h	1 MHz	46.86	124.34 MHz	0.008
900°C/4 h	1300°C/2 h	100 MHz	38.66	23059 MHz	0.0043
900°C/4 h	1300°C/2 h	1 GHz	40.22	6.75 GHz	0.1481
900°C/4 h	1300°C/2 h	1.4 GHz	43.52	4.186 GHz	0.4149
900°C/4 h	1300°C/2 h	1.8 GHz	67.77	3.13 GHz	0.5847
900°C/4 h	1300°C/2 h	2 GHz	51.2	1.96 GHz	1.018

Note: C.T=Calcination Temperature, S.T=Sintering Temperature, f=Frequency, εr=Dielectric Constant, Qf=Quality Factor, tan (σ)=Tangent Loss or Dielectric Loss

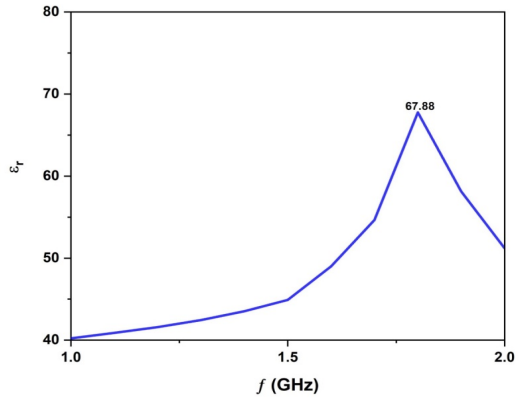

Figure 4. Dielectric constant (εr) of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sintered ceramics at 1300°C.

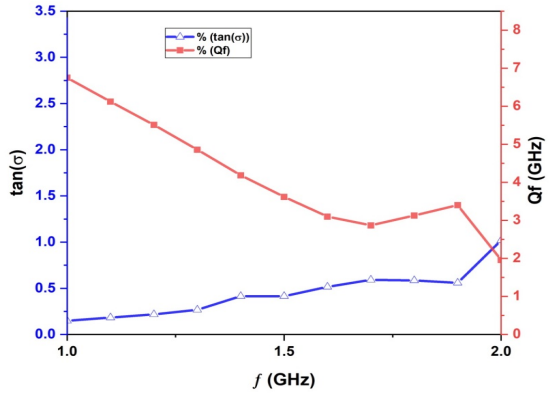

Figure 5. Qf and tan (δ) of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sintered ceramics at 1300°C. **Note:** (—■—)% (Qf), (—△—)%(tan(σ))

Conclusion

The structural and microwave dielectric properties of $(Ba_{0.4}Ca_{0.6})Ti_4O_9$ sintered ceramics at 1300°C were studied. It is found that the εr and Q × f values improved with Ca^{2+} contents. The $((Ba_{0.4}Ca_{0.6})Ti_{4O9}$ sintered ceramics sample had the best εr and Q × f values. Outstanding microwave dielectric properties of εr ~ 67.88 and Q × f ~ 23059 MHz were obtained for $(Ba_{0.4}Ca_{0.6})Ti_{4O9}$ sintered ceramics at 1300°C for 2 hrs, making it a favorable applicant material for microwave wireless communication systemsDensification of CaP ceramics by conventional sintering is an energy-intensive and time-consuming operation. Due to the thermal instability of CaP, treatment at a high temperature for a long time leads to the formation of secondary phases .

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