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Microwave characterisation of $BaCe_2Ti_5O_{15}$ and $Ba_5Nb_4O_{15}$ ceramic dielectric resonators using whispering gallery mode method

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Abstract

A microwave dielectric ceramic resonators based on $BaCe_2Ti_50_{15}$ and $Ba_5Nb_4O_{15}$ have been prepared by conventional solid state ceramic route. The dielectric resonators (DRs) have high dielectric constant 32 and 40 for $BaCe_2Ti_5O_{15}$ and $Ba_5Nb_4O_{15}$, respectively. The whispering gallery mode (WGM) technique was employed for the accurate determination of the dielectric properties in the microwave frequency range. The $BaCe_2Ti_5O_{15}$ and $Ba_5Nb_4O_{15}$ have quality factors $(Q \times F)$ of 30,600 and 53,000 respectively. The quality factor is found to depend on the azimuthal mode numbers. The temperature coefficient of resonant frequency (τ_f) of $BaCe_2Ti_5O_{15}$ and $Ba_5Nb_4O_{15}$ have been measured accurately using different resonant modes and are $+41$ and $+78$ ppm/K, respectively.

Keywords: Dielectric materials; Dielectric resonators; Microwave ceramics; Oxidic ceramics; Microwave resonators

1. Introduction

The recent rapid expansion of telecommunication systems demands dielectric resonators (DRs) as basic components for designing filters and oscillators. Reducing the size is very important for high quality and low cost devices. One of the most suitable ways of miniaturising the filters and reducing their cost is to use high dielectric constant and low loss ceramic DRs. The important characteristics required for a DR are high ε_r for miniaturisation, high Q for selectivity and low temperature variation of the resonant frequency for stability.

Dielectric resonators are normally operated using $TE_{01\delta}$, $TM_{01\delta}$, or $HE_{11\delta}$ mode. The measured quality factors of these modes depend, not only on the material loss tangent, but also on the conducting plates, cavity, radiation losses, etc. Hence, simple measurement of quality factor by the Courtney or stripline methods [1–3] is not sufficient to determine accurately the dielectric loss of the ceramic. How-

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ever, it has been established recently that whispering gallery mode (WGM) modes are necessary to obtain the high level of dielectric field confinement, which, in turn, give rise to low loss tangents for DRs at microwave frequencies [5–9]. The advantages of the WGM method are: their dimensions are large even in millimeteric wavelength band and their quality factors are large. The quality factor of WGM DRs is limited only by the intrinsic losses in the dielectric material. The radiation and conductor losses are negligible and the quality factor $Q \sim 1/\tan\delta$. They also offer good suppression of spurious modes that leak out of the resonator and can be absorbed without perturbing the desired ones [6,8,9]. A survey of literature shows that the WGM method has been used mainly to estimate the Q of sapphire singe crystals [6–9]. It has been reported that WGM resonators exhibit very high Q-factors in the microwave frequency range [6–9]. In WGM resonators, most of the electromagnetic energy is confined to the dielectric near the perimeter of the air–dielectric interface, which, in turn, reduces the radiation and conductor losses. Such high Q-modes offer the possibility of the realisation of high spectral purity sources, operating directly in the microwave domain. Recently, $Ba_5Nb_4O_{15}$ has been reported [10–12] as a high Q DR material with ε_r as 40. The τ_f of Ba_5 Nb_4O_{15} in the literature is found [10–12] to be inconsistent. Hence, a detailed investigation was made to study the microwave dielectric properties using the WGM method. We also report the preparation and characterisation and microwave dielectric properties of a new material $BaCe_2Ti$ $_50_{15}$. We used WGM measurement technique for the accurate determination of the loss tangent and temperature coefficient of $Ba_5Nb_4O_{15}$ (B_5N_4) and $BaCe_2Ti_5O_{15}$ (BC_2T_5) microwave ceramic DRs.

2. Experimental

The DR samples were prepared by the conventional solid state ceramic route. Stoichiometric amount of high purity (99.9% pure) carbonates/oxides were weighed and wet-mixed in distilled water for about an hour and then dried. The B_5N_4 samples were calcined at 1250°C, whereas the BC_2T_5 samples were calcined at 1200°C in a platinum crucible for 4 h. The calcined powders were ground again for 1 h. A 5% polyvinyl alcohol (PVA) solution was added to the powder and the slurry was dried. The powders were again ground well and then pressed uniaxially in tungsten carbide (WC) die of 11-mm diameter. Cylindrical compacts of different thickness in the range 4–8 mm were made under a pressure of 175 MPa. These compacts were sintered in air for 4 h. The sintering temperature of B_5N_4 was 1425°C and that of BC_2T_5 was 1260°C. The density of the compacts were measured by the Archimedes method.

The dielectric constants (ε_r) of these ceramics were measured using an HP 8510 B Network Analyser, coupled with accessories and controlled by an HP 9000, 300 computer. The permittivities were obtained from the TE_{011} resonance mode of the end shorted samples placed between two conducting plates using the method of Hakki and Coleman [1] and modified by Courtney [2]. The unloaded quality factor was measured using both stripline, as well as WGM methods. The Khanna and Garault [3] method was used for Q measurements at lower frequencies. The ceramics were coupled to a microstripline in a brass enclosure. The unloaded quality factors were found from the response of the band rejection filters.

To determine the loss tangent in the WGM ceramic, cylindrical pucks of 10 mm in diameter and 5–7 mm in height were used. A schematic diagram of the copper cavity used for the WGM measure-

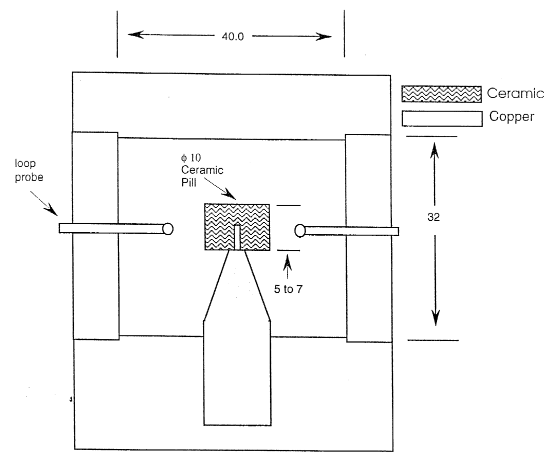

Fig. 1. Schematic diagram of a Cu cavity used for the WGM measurements of ceramic resonators.

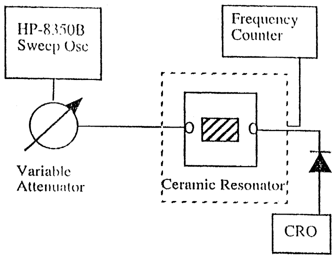

Fig. 2. Schematic diagram of the experimental set-up used for the WGM measurements.

ments is shown in Fig. 1. The copper post used for holding the ceramic puck was designed to minimise the losses. The loss tangents were measured in the 8–24 GHz region, using the reflection configuration. Loop probes were used to excite the resonator. An HP 8350B sweep oscillator was used for the measurements. A microwave frequency counter was used to read out the frequency. The resonant frequencies were determined by the frequency of minimum reflection, and the loaded Q-factor (Q_L) was determined by measuring the half power bandwidth. The coupling was measured using an HP X 382A variable attenuator. The unloaded quality factor was calculated from the coupling factor ~ 3, using the equation $Q_0 = Q_L(1+\beta)$. The schematic diagram of the experimental set-up is shown in Fig. 2.

For the temperature coefficient of resonant frequency (τ_f), the sample was mounted inside a vacuum can and a peltier circuit was used for varying the temperature. Platinum and germanium thermocouples were used to read out and control the temperature inside the can from -14°C to 47°C. The variation in the resonant frequency of the different modes were noted as a function of temperature.

3. Results and discussion

The BC_2T_5 and B_5N_4 ceramics were sintered to form dense ceramics. The measured density of B_5N_4 was 95%. The density of BC_2T_5 was 5.1 g/cm^3 and the percentage density could not be calculated since the structure and symmetry of the unit cell is yet to be determined. Table 1 gives the d-spacing and intensities of the XRD pattern recorded from powdered sample BC_2T_5 and Fig. 3 shows a typical micrograph. The SEM picture shows that the grains are up to 4 μm in size and no secondary phase is observable. The details of preparation and material

Table 1
X-ray powder diffraction data for $BaCe_2Ti_5O_{15}$

d	2θ	I (real)
4.43898	19.986	16.40
3.78960	23.456	13.08
3.34766	26.606	14.64
3.24191	27.491	16.96
3.11830	28.603	100.00
3.04290	29.328	18.30
2.93067	30.477	12.41
2.76576	32.343	14.36
2.70041	33.148	34.93
2.65352	33.751	11.40
2.48203	36.161	11.00
2.28990	39.314	9.84
2.22594	40.492	12.05
2.12852	42.433	12.93
2.09180	43.215	12.59
2.03775	44.422	9.67
1.95557	46.395	8.92
1.90898	47.596	45.41
1.82534	49.922	8.92
1.75772	51.983	8.89
1.67106	54.899	7.69
1.63897	56.067	8.11
1.62814	56.474	34.74
1.60557	57.340	8.39
1.59116	57.909	9.41
1.55889	59.225	10.99
1.43237	65.065	8.76
1.42042	65.681	8.25
1.35012	69.576	11.99

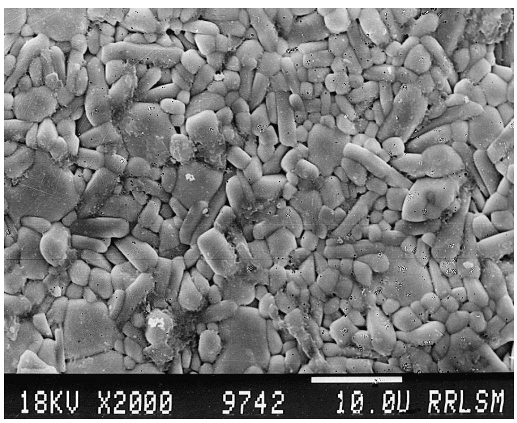

Fig. 3. SEM picture recorded from $BaCe_2Ti_5O_{15}$ ceramic.

Table 2
Comparison of the quality factors of $BaCe_2Ti_5O_{15}$ and $Ba_5Nb_4O_{15}$ using the stripline and WGM methods

Material	Dielectric constant	$Q_u \times F$ (in GHz; WGM method)	$Q_u \times F$ (in GHz; stripline method)	T_f
$BaCe_2Ti_5O_{15}$	32	1780×17.19	3570×5.355	41
$Ba_5Nb_4O_{15}$	40	3300×16.012	4140×4.4789	78

characterisation of B_5N_4 are given in Ref. [10]. The XRD and SEM studies reveal the absence of secondary phase in B_5N_4. The microwave dielectric properties, such as dielectric constant and temperature variation of resonant frequency, were measured using the end shorted method and unloaded quality factor using the stripline method. The B_5N_4 ceramic has a dielectric constant of 40 and unloaded quality factor of 4140 at 4.4789 GHz. The density corrected dielectric constant for B_5N_4 is 42. The BC_2T_5 ceramic has a dielectric constant of 32 and unloaded quality factor of 3570 at 5.355 GHz and T_f of 41 as given in Table 2.

It is common to denote a mode with a dominant axial electric field dependence as an E mode (quasi-TM) and a dominant magnetic field dependence as an H-mode (quasi-TE). Even though the material is isotropic, we implemented anisotropic software [4] to determine the hybrid nature of the modes under investigation. The resonant frequencies were calculated from the dimensions and ε, determined by the end shorted method assuming $\varepsilon_{\parallel} = \varepsilon_{\perp}$, where $\varepsilon_{\parallel}$ is the permittivity parallel to the anisotropic axis, and $\varepsilon_{\perp}$ is the permittivity perpendicular to the anisotropic axis. The electric energy filling factors for E and H modes parallel, $P_{e\parallel}$, and perpendicular, $P_{e\perp}$, are from the following equation:

$$P_{e\perp} = 2\frac{\partial f}{\partial \varepsilon_{\perp}}\frac{\varepsilon_{\perp}}{f}$$

$$P_{e\parallel} = 2\frac{\partial f}{\partial \varepsilon_{\parallel}}\frac{\varepsilon_{\parallel}}{f}$$

For quasi-TE mode, the energy will be mainly stored in the perpendicular direction and $P_{e\perp} > P_{e\parallel}$, while for a quasi-TM mode, the energy will be mainly stored in the parallel direction and $P_{e\parallel} > P_{e\perp}$.

The dielectric loss tangent for the isotropic dielectric can be solved [9] using the following equation:

$$Q_{(E)}^{-1} = \tan\delta(P_{e\perp} + P_{e\parallel}) + R_S/G_{(E)}$$

$$Q_{(H)}^{-1} = \tan\delta(P_{e\perp} + P_{e\parallel}) + R_S/G_{(H)}$$

where R_S is the surface resistance of the cavity enclosing the DRs. The conductor losses decrease as the surface resistance becomes smaller and as the geometric factor increases [9]. Further, the conductor losses were minimised by keeping the samples away from the conducting walls in the present experiments. For WGMs, the geometric factor G is significantly large that the effect of the cavity can be

Table 3
Measured and predicted frequencies and corresponding filling factors of quasi-TM modes of $Ba_5Nb_4O_{15}$

m	Quasi-TM modes	Frequency measurement (GHz)	Frequency measurement (GHz)	Electric energy filling factor $P_{\perp}$	Electric energy filling factor $P_{\parallel}$	Electric energy filling factor (total)	Q_u
2	TM_{210}	–	7.185	0.462	0.5307	0.9927	–
3	TM_{310}	8.928	8.952	0.3286	0.6679	0.9965	5600
4	TM_{410}	10.725	10.693	0.2427	0.7553	0.998	2750
5	TM_{510}	12.493	12.47	0.1856	0.8132	0.9987	2750
6	TM_{610}	14.253	14.227	0.1462	0.853	0.9991	2700
7	TM_{710}	16.012	16.009	0.1215	0.879	1	3300
8	TM_{810}	19.07	18.59	0.0998	0.9006	1	–

Table 4
Measured and predicted frequencies and corresponding filling factors of quasi-TM modes of $BaCe_2Ti_5O_{15}$

m	Quasi-TM modes	Frequency measurement (GHz)	Frequency measurement (GHz)	Electric energy filling factor $P_{\perp}$	Electric energy filling factor $P_{\parallel}$	Electric energy filling factor (total)	Q_u
2	TM_{210}	7.612	7.973	0.4338	0.5596	0.9935	–
3	TM_{310}	9.542	9.499	0.3035	0.6934	0.9969	3000
4	TM_{410}	11.472	11.124	0.2218	0.7764	0.9982	2975
5	TM_{510}	13.39	13.02	0.1685	0.8304	0.9989	1550
6	TM_{610}	15.3	15.15	0.1321	0.8672	0.9993	1490
7	TM_{710}	17.19	17.27	0.1063	0.8932	0.9995	1780
8	TM_{810}	19.08	18.59	0.0873	0.9123	0.9996	–

ignored when compared to the loss tangent. Hence, the calculations were done neglecting the term containing R_S. The electric energy filling factors of DR for all these modes are close to unity (Tables 3 and 4). The modes, which have high electric energy filling factor have high Q and are the WGM modes. It may be noted that there are several other modes in the frequency range 8–20 GHz, which are difficult to identify experimentally since they are very sensitive to the surrounding when kept outside the cavity. Hence, the calculated and experimental results for such modes are not indicated in Tables 3 and 4.

The WGM mode microwave measurements of these ceramics were performed for the accurate determination of Q_u and the results are compiled in Table 2. The measurements were made in the reflection configuration using the Cu cavity as shown in Fig. 2. For details of mode identification and measurement the reader is referred to Ref. [13]. The X-band quasi-T_{mnp} resonant mode families with azimuthal mode numbers m from three to eight were identified for B_5N_4 and BC_2T_5 ceramics. The mode families were identified by the excitation and observation of the $H\varphi$-field, using two magnetic loop probes in a radial plane. The mode identification was made by keeping the sample outside the cavity in air and the measurements were in transmission with one probe stationary and the other probe moved in the azimuthal, axial and radial directions. The mode identification was done by keeping the sample outside the cavity, since there will be a small change in the resonant frequency, which may cause difficulty in identifying the modes and comparing with the predicted ones, and also to avoid confusion with cavity modes. This is because the resonant frequency (predicted) was calculated for the sample alone and not inside the cavity (without the small effect due to the cavity), after identifying the modes, the sample was kept inside the cavity and Q was measured for the different modes. For each mode, the unloaded quality factor (Q_u) was accurately determined from a measurement of loaded Q and the corresponding coupling.

The frequencies of the different modes were theoretically predicted using the method described in Ref. [8]. It can be seen from Fig. 4 and Table 3 that the predicted and measured quasi-TM modes are in good agreement for B_5N_4. The quality factor of this

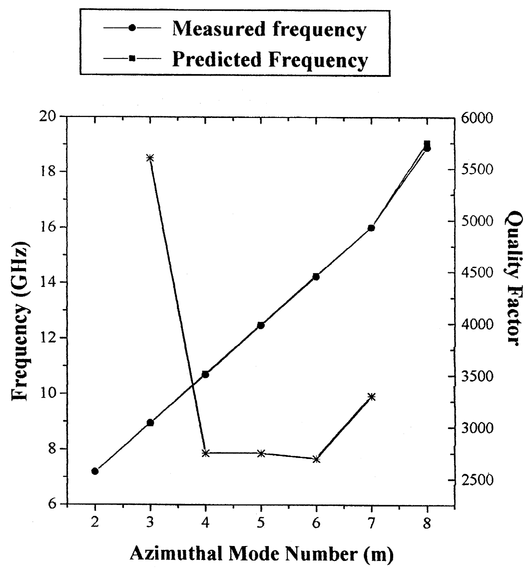

Fig. 4. Predicted and measured frequencies and quality factor of $Ba_5Nb_4O_{15}$ ceramic with respect to the azimuthal mode number (m).

resonator is 5600 at 8.92 GHz for the TM_{310} WGM mode. It is evident from the figure that, as the azimuthal mode number increases, the quality factor decreases and after a certain limit ($m = 6$), it shows a small increase. The observed high quality factor at low azimuthal mode numbers is a clear indication of the effect of cavity on the resonator quality factor. The quality factor of the empty cavity is higher than the ceramic. Thus, for small azimuthal numbers, the resonator exhibits a higher value of Q due to the cavity effect. The quality factor is 3300 at 16.012 GHz for azimuthal mode number 7 and $Q \times F = 53{,}000$, which is much higher than earlier reports [10,12] for this material.

In BC_2T_5 also, a cavity has pronounced influence on the quality factor of the resonator at low azimuthal mode numbers (see Fig. 5). As the azimuthal mode number increases, Q decreases initially. This decrease is due to the decreasing effect of the cavity. As the azimuthal mode number increases further, the quality factor increases. A study of Table 4 shows that as the azimuthal mode number increases, the electric energy filling factor also increases. But a study of measured Q shows that it initially decreases and then increases. The initial high Q is due to the effect of cavity. The quality factor of BC_2T_5 at 17.19 GHz is 1780 or $Q \times F$ is 30,000 for the TM_{710} mode. The $Q \times F$ measured at 5.335 GHz by the stripline method is 19,000.

The temperature variation of resonant frequency of these ceramics were measured from 259 to 320 K. The T_f obtained for different modes gave consistent value of 78 ppm/K for B_5N_4 ceramic, in agreement with the earlier report of Vineis et al. [11]. We have measured eight modes in the 8–18 GHz region. The T_f obtained for most of the modes were around 78 ppm/K (Fig. 6). A linear variation of temperature coefficient of resonant frequency with respect to different resonant frequencies can be seen in BC_2T_5

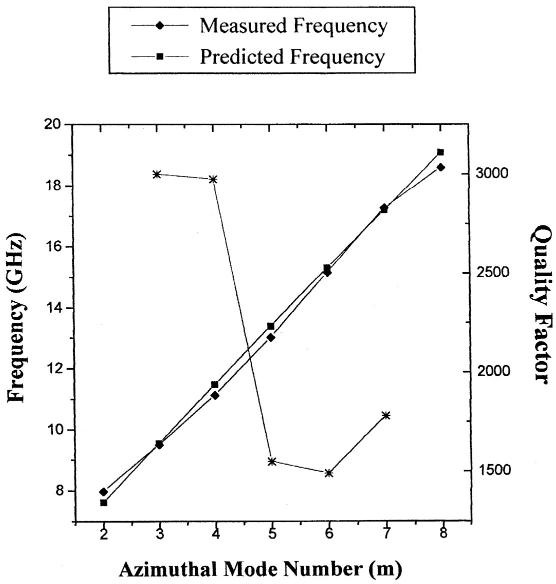

Fig. 5. Predicted and measured frequencies and quality factor of $BaCe_2\ Ti_5O_{15}$ ceramic with respect to the azimuthal mode number (m).

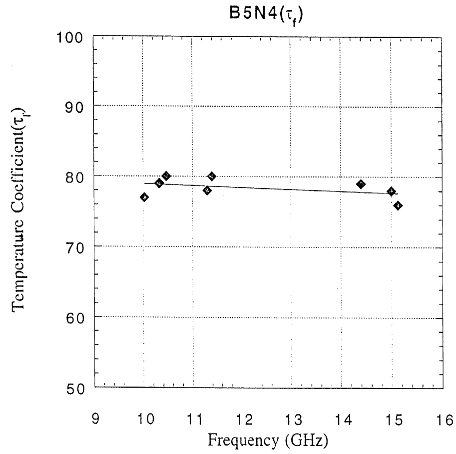

Fig. 6. Temperature coefficient of $Ba_5Nb_4O_{15}$ ceramic at different resonant frequencies.

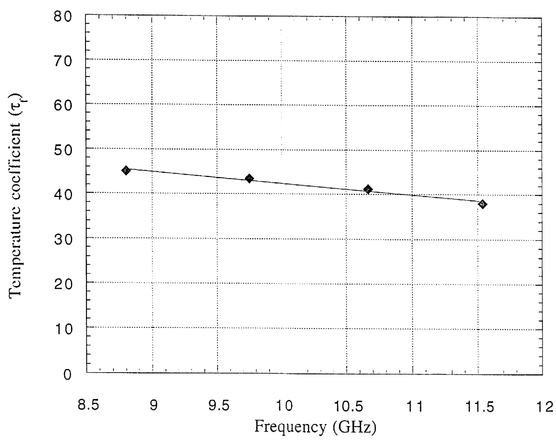

Fig. 7. Temperature coefficient of $BaCe_2\ Ti_5O_{15}$ ceramic at different resonant frequencies.

ceramics (Fig. 7). Microwave dielectric properties of BC_2T_5 is being reported for the first time.

4. Conclusion

The WGM method has been used to measure the dielectric properties of $Ba_5Nb_4O_{15}$ and $BaCe_2Ti_5O_{15}$ ceramic DRs. The DRs have dielectric constants of 32 and 40 for $BaCe_2Ti._50_{15}$ and $Ba_5Nb_4O_{15}$, respectively. A better understanding of the behaviour of frequency on quality factor has been established. The highest quality factors $(Q \times F)$ were 53,000 at 16 GHz for $Ba_5Nb_4O_{15}$ and 30,600 at 17.19 GHz for $BaCe_2Ti_5O_{15}$. Predicted and measured frequencies show good agreement in $Ba_5Nb_4O_{15}$ resonator. We obtained a temperature coefficient T_f of ~ 78 ppm/°C for $Ba_5Nb_4O_{15}$ and ~ 41 ppm/°C for $BaCe_2Ti_5O_{15}$.

Acknowledgements

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