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Dielectric properties and mixture behavior of y$(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-(1-y)$CaTiO_3$ ceramic system at microwave frequency

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ABSTRACT

The microstructures and the microwave dielectric properties of the y$(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-(1-y)$CaTiO_3$ ceramic system were investigated. In order to achieve a temperature coefficient of resonant frequency (τ_f) stable material, $CaTiO_3$ (τ_f ~ +800 ppm/°C) was chosen as a τ_f compensator and added to $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ (τ_f ~ -59 ppm/°C) to form a two phase system. It was confirmed by the X-ray diffraction (XRD) and Energy dispersive X-ray (EDX) analysis. Although dielectric constant (ε_r) of the specimen could be boosted by increasing amount of $CaTiO_3$, it would instead render a decrease in the quality factor (Q × f). The τ_f value is strongly correlated to the compositions and can be controlled through the existing phases. By appropriately adjusting the y-value in the y$(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-(1-y)$CaTiO_3$ ceramic system, near-zero τ_f value can be achieved. A new microwave dielectric material, 0.4$(Mg_{0.95}Co_{0.05})_4Ta_2O_9-0.6CaTiO_3$ applicable in microwave devices are suggested and possesses the dielectric properties of a dielectric constant ε_r ~ 25.78, a Q × f value ~200,000 GHz and a τ_f value ~ -4.69 ppm/°C. It is proposed as a very promising dielectric material for low-loss microwave and millimeter wave applications.

1. Introduction

Owing to the rapid development of microwave communication, the microwave dielectric ceramics have attracted the increasing interests because of their important applications in microwave devices such as filters, resonators, antennas and other key components for decades [1–7]. The main driving force for its application could ascribe to the extremely necessitate low dielectric loss (high quality factor, $Q = 1/\tan\delta$), smaller device size (high dielectric constant, ε_r), temperature stability (near-zero temperature coefficient of resonant frequency, τ_f) and low cost.

In general, high dielectric constant materials exhibit high dielectric loss (low Q × f value), while low loss ceramics are usually accompanied by low ε_r value. A number of materials with excellent dielectric properties have been reported for microwave applications. Among them corundum-structured ceramics with the general formula $A_4B_2O_9$ (where $A = Mg^{2+}$, Zn^{2+}, Co^{2+}, and Ni^{2+}; $B = Ta^{5+}$ and Nb^{5+}) have attracted much attention because of their high Q values and low-dielectric constants [8–13]. For instance, $Mg_4Nb_2O_9$ was investigated to exhibit excellent microwave dielectric properties by Yoshida [14] et al. (ε_r ~ 12.4, $Q \times f$ ~ 192,000 GHz and τf ~ −70.5 ppm/°C). Further research was accomplished to promote its $Q \times f$ to 247,000 GHz by partially substituting Mg with Zn to form a solid solution [15]. In particularly, $Mg_4Ta_2O_9$, belonging to trigonal crystal system with a space group of *P*3 *cl*, has been widely used in microwave ceramic capacitors and resonators because of its high Q value and thermal stability [16].

It exhibits microwave dielectric properties with a high $Q \times f$ value of 345,000 GHz, a dielectric constant ε_r ~ 10, and a temperature coefficient of resonant frequency τf ~ −70 ppm/°C [8–13]. Many papers reported on the effect of B-site substitution of $A_4B_2O_9$ ceramics [8–13], whereas were only few dealing with A-site replacement [8–13]. Moreover, small substitution has been shown to have significant effect on the improvement of $Q \times f$ value for dielectric ceramics [17,18] and also, Zn^{2+} (0.74 Å, CN = 6) and Mg^{2+} (0.72 Å, CN = 6) has a similar radius [19]. These provided us the motive to study the effect of partially substituting Mg with Zn on the microwave dielectric properties of $Mg_4Ta_2O_9$ ceramics.

According to previous researches, $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$

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possesses high dielectric constant ε_r ~12.28, high Q × f value ~440,000 GHz and negative τ_f value −59 ppm/°C. However, the $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ ceramics has a negative τ_f value, which is not suitable for practical use. In this work, $CaTiO_3$ (ε_r ~140, Q × f value ~3600 GHz, τ_f ~800 ppm/°C) [20,21] have been individually employed as an effective τ_f compensator for materials.

The dielectric properties at microwave frequencies of the sintered ceramics were characterized and discussed in terms of the densification and the compositional ratio of the specimens. In addition, the X-ray diffraction (XRD) patterning and scanning electron microscopy (SEM) analysis were also employed to study the crystal structures and microstructures of the ceramics.

2. Experimental procedures

$(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ and $CaTiO_3$ ceramic powders were prepared using the solid−state reaction method by mixing individual high-purity oxides MgO, CoO, Ta_2O_5, and $CaCO_3$, TiO_2. The starting materials were stoichiometrically weighed after MgO was sintered at 800 °C for 6 h to remove moisture and carbonates. The powders were then dry-mixed with an agate mortar and pestle and subsequently wet-mixed using distilled water. The prepared powders were dried and calcined at 1100 °C for 4 h in air. The calcined powders were mixed according to the molar fraction $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ and then re-milled for 24 h. The calcined powder with the organic binder polyvinyl alcohol was pressed into pellets using a uniaxial press. The binder was then evaporated at 650 °C for 12 h. Sintering were carried out at 1325–1425 °C for 4 h in air. The powder and bulk X-ray diffraction (XRD, Rigaku D/Max III.V) patterns were collected using CuKα radiation (at 30 kV and 20 mA) and a graphite monochrometer in the 2θ range of 10–60°. The microstructural observations and analysis of the sintered surface were performed using scanning electron microscopy (SEM, Philips XL-40FEG).

The bulk densities of the sintered pellets were measured using the Archimedes method. Microwave dielectric properties such as the electric permittivity and unloaded Q were measured at 6–12 GHz using the post-resonance method as suggested by Hakki and Coleman [22,23]. This method uses parallel conducting plates and coaxial probes in TE_{011} mode, where TE means transverse electric waves, the first two subscript integers denote the wave guide mode, and the third integer denotes the order of resonance in an increasing set of discrete resonant lengths. The temperature coefficient of resonant frequency was measured in the temperature range of 20–80 °C. A system that combines an HP8757D network analyzer and an HP8350B sweep oscillator was employed in the measurements.

3. Results and discussion

Microwave dielectric properties of $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramic system sintered at 1375 °C for 4 h are illustrated in Table 1. The τ_f is a function of y owing to a variation in its compositional ratio. Moreover, a near-zero τ_f can be achieved using $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics. Fig. 1 shows the room temperature XRD patterns recorded for $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramics sintered at 1375 °C for 4 h. Significant variation was not spotted from the X-ray diffraction patterns of the ceramic system at different y values. No second phase was detected. Moreover, it is understood that crystal structures of $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ and $CaTiO_3$ are hexagonal (ICDD-PDF #00-036-1381) and cubic (ICDDPDF#00-040-1500), respectively. The XRD patterns showed a two-phase system with a $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ phase. $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramics showed a mixture of a main phase $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ and a minor phase $CaTiO_3$ as increasing $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ content.

Fig. 2 shows the XRD patterns of the $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramic sintered at various temperatures for 4 h. The XRD patterns showed that peaks indicating the presence of $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ as the main crystalline phase. According to the XRD patterns, the $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ phase exists in these specimens. The X-ray diffraction patterns of the $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramic systems have not been changed significantly with sintering temperatures in the range 1325–1425 °C. The formation of mixed phases in the $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics is due to structural differences; therefore, a solid solution could not be obtained.

Fig. 3 shows the SEM of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics at different temperatures for 4 h. The $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics was not dense and the grain did not grow below 1375 °C. Porous microstructures were observed at 1325 °C and 1350 °C highlighted by red circle. However, abnormal grain growth was observed at 1425 °C (as marked in Fig. 3(f)), which might degrade the microwave dielectric properties of the ceramics.

Energy dispersive X-ray (EDX) analysis was used in combination with scanning electron microscopy to distinguish every grain for $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics sintered at 1375 °C as shown in Fig. 4(a). The EDX datum and data of corresponding spots A and B were showed in Fig. 4(b). The grain morphology of well developed, $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics could be grouped into two types: large grains (Spot A) were $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$, small cubic-shape grains (Spot B) were $CaTiO_3$. It revealed that the surface of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics had Mg, Co, Ta, Ca, Ti, and O ions. And the EDX evidences were in agreement with XRD results that can be obtained

Table 1
Microwave dielectric properties of y(Mg0.95Co0.05)4Ta_2O_9-(1-y)$CaTiO_3$ ceramic system sinter at 1375 °C for 4 h.

Y	Bulk density (g/cm^3)	ε_r	Q × f(GHz)	τ_f(ppm/°C)
0.2	4.82	47.32	48,058	77.74
0.4	5.53	25.78	201,297	−4.69
0.5	5.62	22.44	256,335	−10.71
0.6	5.82	20.35	309,636	−13.35
0.8	5.98	15.28	390,542	−34.89

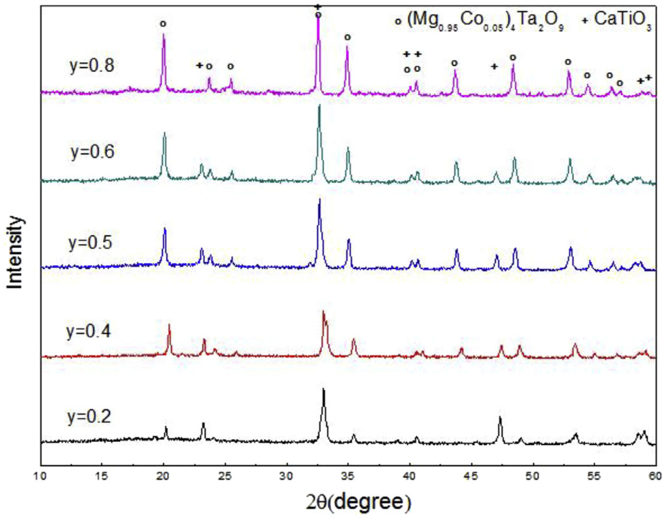


Fig. 1. X-ray diffraction patterns of $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramics sintered at 1375 °C for 4 h.

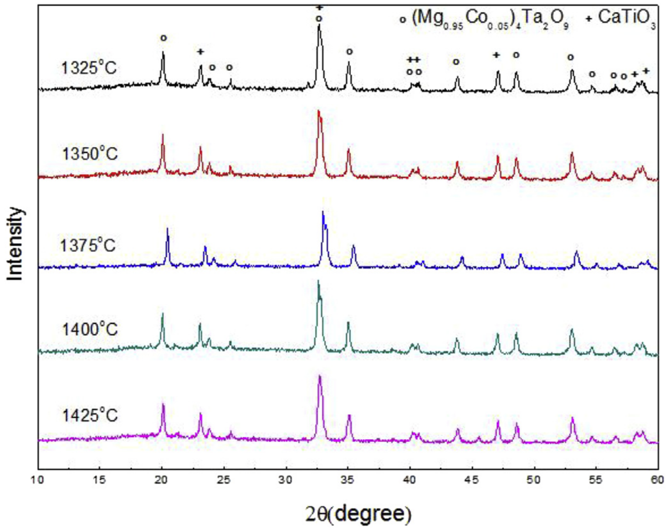
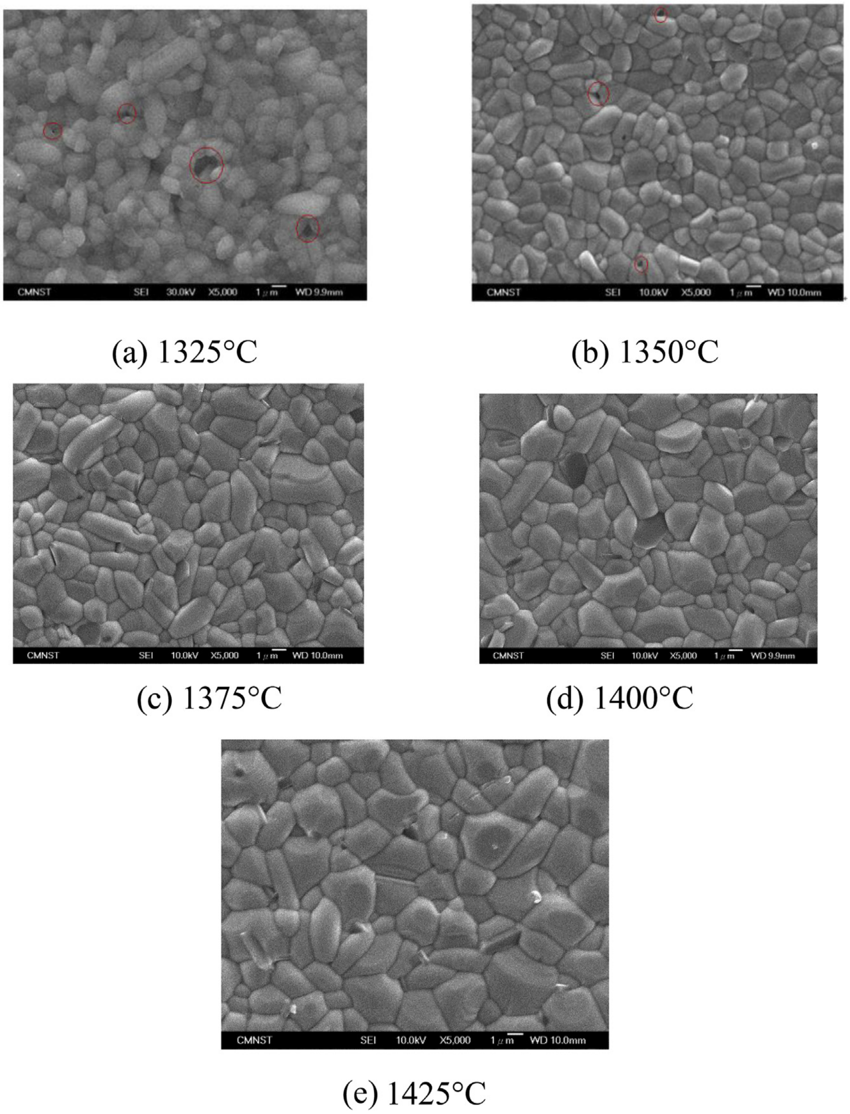


Fig. 2. X-ray diffraction patterns of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics sintered at various temperatures for 4 h.

$0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics.

Fig. 5 shows the apparent densities of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics sintered at various sintering temperatures for 4 h. With increasing sintering temperature, the apparent density of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramic was increased to a maximum value of 5.45 g/cm^3 at 1375 °C and thereafter it decreased. Moreover, the degradation of apparent density at temperatures above 1375 °C was owing to the abnormal grain growth as observed in Fig. 3.

The dielectric constant and Q × f values of $0.4(Mg_{0.95}Co_{0.05})_4$-$Ta_2O_9$-$0.6CaTiO_3$ ceramics at various sintering temperatures for 4 h are shown in Fig. 6. Variation of dielectric constant was consistent with that of density. The dielectric constant of $0.4(Mg_{0.95}Co_{0.05})_4$-$Ta_2O_9$-$0.6CaTiO_3$ ceramic initially increased with increasing sintering temperature. After reaching the maximum at 1375 °C, it decreased. Maximum ε_r value of 25.8 was obtained for $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics sintered at 1375 °C for 4 h. According to Lichteneker rule [24], it indicates further increase in the sintering temperature does not certainly lead to a higher dielectric constant. Because, general Lichterecker mixing rule can be written as:



(a) 1325°C

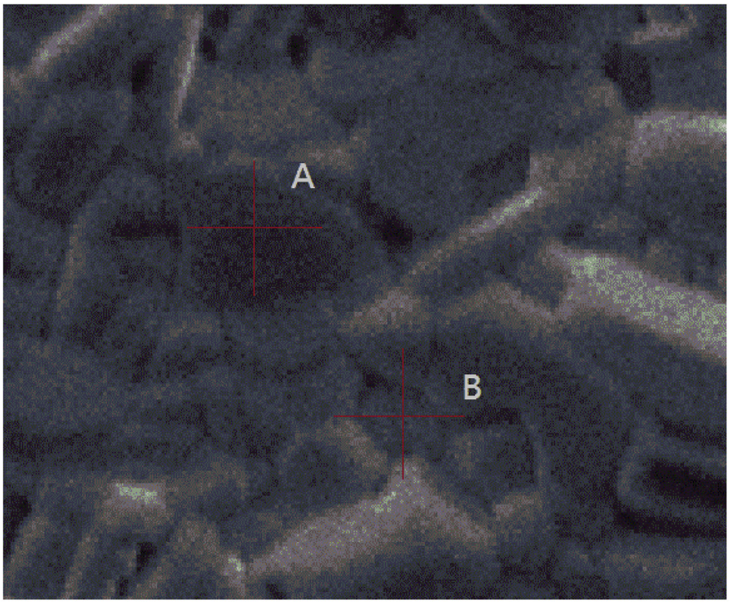
(b) 1350°C

(c) 1375°C

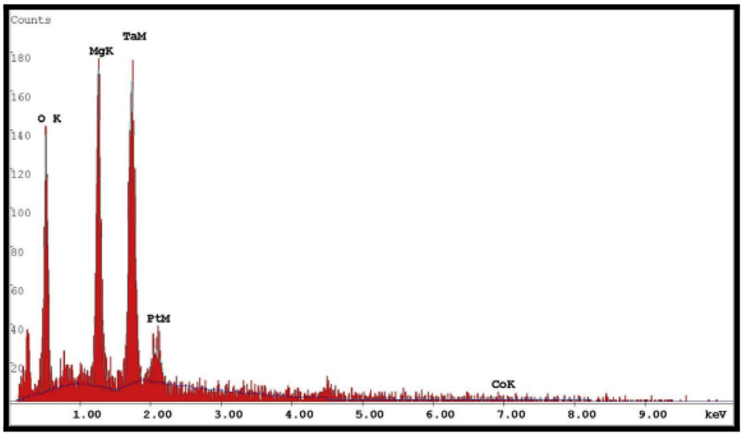
(d) 1400°C

(e) 1425°C

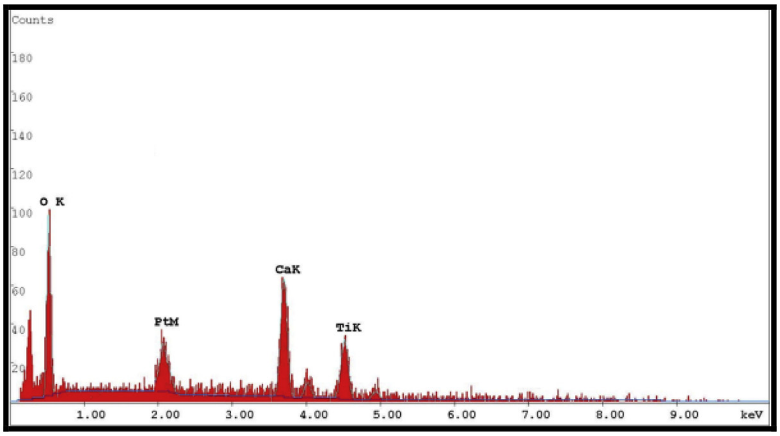
Fig. 3. SEM photographs of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics (a) 1325, (b) 1350, (c) 1375 (d) 1400, (e) 1425 °C sintered at various temperatures for 4 h.



(a)



Spots A: Mg K:57.94, Co L:1.77, Ta L:16.14, Ca K: 0, TiK:0, O K:51.66



(b)

Spots B: Mg K:0, Co L:0, Ta L:0, Ca K: 18.87, TiK: 19.57, O K:58.43

Fig. 4. (a) The marks of SEM for the 0.4($Mg_{0.95}Co_{0.05}$)$_4$$Ta_2O_9-0.6CaTiO_3$ ceramics sintered at 1375 °C; (b) EDX datum of 0.4($Mg_{0.95}Co_{0.05}$)$_4$$Ta_2O_9-0.6CaTiO_3$ ceramics for spots A and B.

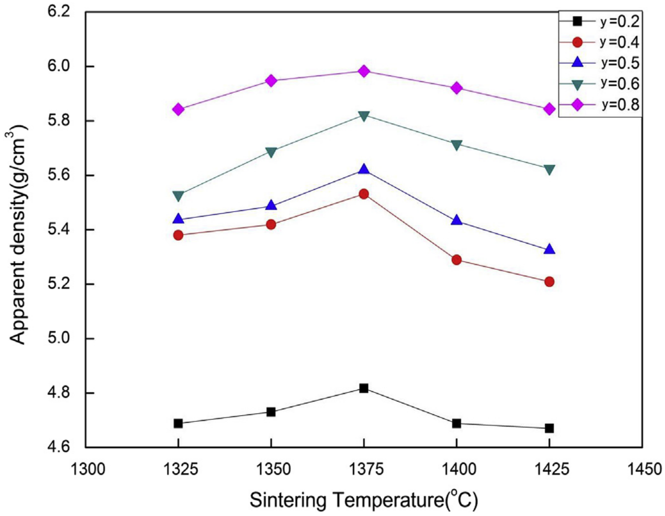


Fig. 5. Apparent density of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics system sintered at different temperatures addition.

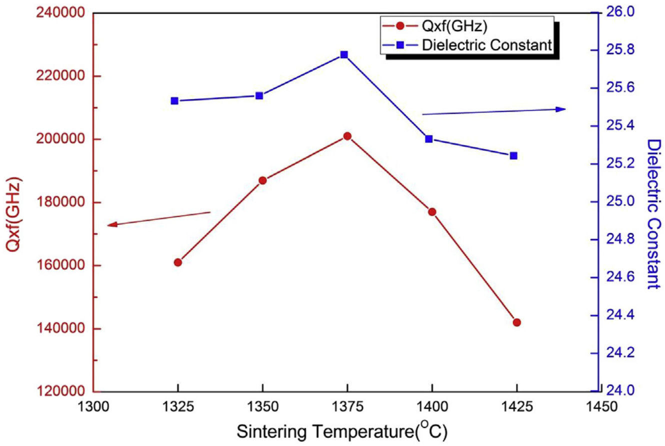


Fig. 6. ε_r and Q × f values of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics system sintered at various temperatures for 4 h.

$$\varepsilon_m^\alpha = V_h\varepsilon_h^\alpha + V_1\varepsilon_l^\alpha \tag{1}$$

where ε_h and ε_l are the relative dielectric constants of the high-dielectric phase and low-dielectric phase respectively, v_h and v_l the volume fractions of the high dielectric phase and low-dielectric phase ($v_h + v_l = 1$), ε_m the effective dielectric constant of the composite, and α a parameter that determines the type of mixing rule.

When $\alpha = -1$, one has a serial mixing rule:

$$\frac{1}{\varepsilon} = \frac{v_h}{\varepsilon_h} + \frac{v_l}{\varepsilon_l} \tag{2}$$

and when $\alpha = 1$, a parallel mixing rule:

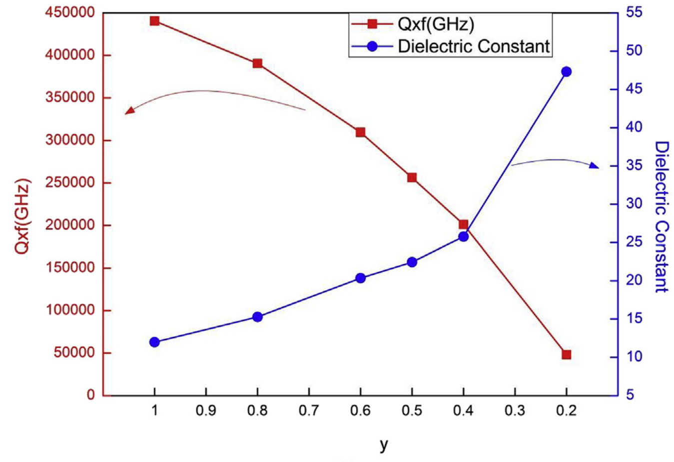
$$\varepsilon_m = V_h\varepsilon_h + V_1\varepsilon_l \tag{3}$$

In the case where $\alpha \rightarrow 0$, Lichterecker proposed an intermediate form between the serial and parallel form called logarithmic mixing rule:

$$\log\varepsilon_m = V_h \log\varepsilon_h + V_1 \log\varepsilon_l \tag{4}$$

The dielectric constant of $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ increased with $CaTiO_3$ apparently increasing. The dielectric constant for $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramics sintered at 1375 °C for 4 h are showed in Table 1. The measured dielectric constant was corrected for porosity [25]. The dielectric constant of $CaTiO_3$ and $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ are 140 and 12.28, respectively. The dielectric constant decreases rapidly as y varies from 0.2 to 0.8. Table 1 shows that the dielectric constant of the system under study follows a mixture-like behaviour as in $(1-x)La(Zn_{1/2}Ti_{1/2})O_3$–$xSrTiO_3$ [26]. Sareni et al. [27] have also reported a similar variation of dielectric constant with composition in the finite element calculations of the two-component dense composites.

Fig. 6 shows the Q × f values of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics at various sintering temperatures for 4 h. The microwave dielectric loss is mainly caused not only by the lattice vibrational modes, but also by the pores, the second phases, the impurities, or the lattice defect [24]. Apparent density also plays an important role in controlling the dielectric loss, and has been shown for other microwave dielectric materials. Since the variation of $Q \times f$ was also consistent with that of density, it suggested the dielectric loss of $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics was



(b)

Fig. 7. (b) ε_r and Q × f values of $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramics system sintered at 1375 °C for 4 h.

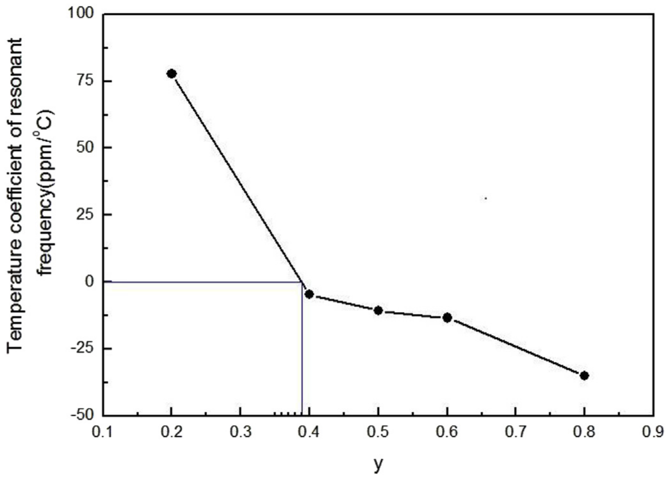


Fig. 8. Temperature coefficient of resonant frequency values of $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramics system sintered at 1375 °C.

mainly controlled by apparent density. Maximum Q × f value of 200,000 GHz was obtained for $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramics sintered at 1375 °C for 4 h. The decrease of Q × f value was attributed to inhomogeneous grain growth resulting in a reduction in density as observed in Fig. 3. The Q × f has the same trend with dielectric constant and bulk density.

Fig. 7 shows the ε_r and Q × f values of $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramics sintered at1375 °C for 4 h. The more $CaTiO_3$ addition, the dielectric constant increased, due to the dielectric constant of $CaTiO_3$ is 140, which is larger than $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$. But the Q × f values were decreased with more $CaTiO_3$ additions, because the Q × f value of $CaTiO_3$ is 3600 GHz is lower than $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$(Q × f = 440,000 GHz).

Fig. 8 shows the temperature coefficients of resonant frequency τ_f of $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramic system sintered at 1300 °C for 4 h with different *y* values. The temperature coefficient of resonant frequency is well known to be governed by the composition, the additives, and the second phase of the materials. Since the τ_f values of $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$and $CaTiO_3$ are −59 and + 800 ppm/°C, respectively, increasing $CaTiO_3$ content makes the τ_f value become more positive. It implies that zero τ_f can be achieved by tuning the amount of $CaTiO_3$ content. In fact, with $y = 0.4$, a nearly zero τ_f value was achieved for $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramic system sintered at 1375 °C for 4 h.

4. Conclusions

The microwave dielectric properties of the $y(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$(1-y)CaTiO_3$ ceramic system were studied. Hexagonal structured $(Mg_{0.95}Co_{0.05})_4Ta_2O_9$ and cubic-structured $CaTiO_3$ were identified by XRD analysis. The microwave dielectric properties are strongly related to the density and the compositional ratio of the ceramics. In comparison with that of $MgTiO_3$–$CaTiO_3$, specimen with $0.4(Mg_{0.95}Co_{0.05})_4Ta_2O_9$-$0.6CaTiO_3$ ceramic system can be sintered at a low temperature 1375 °C and possesses a better combination of microwave dielectric properties: a $\varepsilon_r = 25.78$, a Q × f = 200,000 GHz (measured at 9.1 GHz) and a τ_f value −4.69 ppm/°C. The extremely low-loss dielectric is a very promising material for microwave and millimeter-wave applications.

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