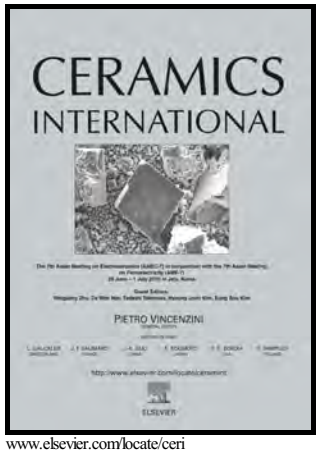

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$MgTiO_3/TiO_2/MgTiO_3$: an ultrahigh-Q and temperature-stable microwave dielectric ceramic through cofired trilayer architecture

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ABSTRACT

A cofired trilayer ceramic architecture showing as $MgTiO_3/TiO_2/MgTiO_3$ was designed to realize temperature-stable and ultrahigh-Q microwave dielectrics in the typical $MgTiO_3$-TiO_2 system. The effects of TiO_2 content on the microwave dielectric properties of cofired trilayer ceramics were studied. Through the design of cofired trilayer architecture, the chemical reactions between $MgTiO_3$ and TiO_2 were limited within a narrow region of $MgTiO_3/TiO_2$ interfaces (~15 μm in width), which could be beneficial for optimizing the microwave dielectric properties. Excellent characteristics of ε_r ~ 18.38, $Q\times f$ value ~ 169 900 GHz and τ_f ~ −1 ppm/°C were gained for the $MgTiO_3/TiO_2/MgTiO_3$ ceramic architectures stacked with 1.63 vol% TiO_2. The current work could serve as new strategies to develop high-performance dielectric resonators and multilayers for 5G wireless communication applications.

Keywords: microwave dielectric ceramic; cofired multilayer architecture; temperature stability; high Q; $MgTiO_3$; TiO_2

1. Introduction

Microwave dielectric ceramics (MWDCs) have been considered the crucial components in modern wireless communication systems and industry, with wide applications in filters, resonators, substrates, *etc* [1-4]. To keep the pace of technological progress, particularly for the transition from 4G to 5G communication, MWDCs are required to have a proper permittivity (ε_r), an ultrahigh $Q \times f$ value (> 100 000 GHz; $Q = 1/\tan\delta$) for good frequency selectivity and a near-zero temperature coefficient of resonant frequency (τ_f) to enable good temperature stability [5-7].

Since "dielectric resonator" (DR) was first predicted in 1939 [1], nowadays MWDCs with diverse performance are all-pervasive, and some of them have gradually realized their commercial applications used as DRs, such as $Ba(Mg,Zn)TaO_3$, $CaTiO_3$-$NdAlO_3$, $(Zr,Sn)TiO_4$ and BaO-Nd_2O_3-TiO_2 [2,4,8,9]. However, it leaves behind many counterparts with large τ_f value at the same time, which makes them far from applicable [2,8]. To develop temperature-stable MWDCs based on current materials, many efforts have been done extensively. Traditionally the idea of tuning τ_f is to introduce foreign materials (known as temperature compensators) with opposite τ_f through conventional solid-state mixed route, such as $CaTiO_3$-$NdAlO_3$ solid solutions and $MgTiO_3$-$CaTiO_3$ composites [2,3,10]. Actually, not all of the introduced temperature compensators can coexist well with the matrix materials yet. The underlying chemical reactions between them would produce some new or secondary phases, which would influence the microwave dielectric properties, particularly making τ_f vary beyond the expected. For instance, TiO_2 is well known for its special microwave dielectric characteristic of a high $\varepsilon_r \sim 105$ and a large positive $\tau_f \sim +450$

ppm/°C. It can serve as an ideal temperature compensator for many MWDCs (including $ZnNb_2O_6$) having a high $Q\times f$ value and a negative τ_f [11-13]. However, in the typical (1-x)$ZnNb_2O_6$-$x$$TiO_2$ system, the chemical reactions between end members seem easy to occur, with respect to the similarity in crystal structure between them. It leads the materials undergo several phase regions along with the increase of TiO_2 content. As a result, a relatively large amount of TiO_2 (x = 0.58) is required to obtain near-zero τ_f and the $Q\times f$ value is as low as 6 000 GHz [12].

Since the possibility of tuning τ_f by stacking two types of cylindrical materials with opposite τ_f was proposed, the attempts of layer stacked engineering have been made to develop temperature-stable MWDCs [14]. As reported, however, most of them use organic glues to bond each layer together, such as $Ba_5Nb_4O_{15}$/5ZnO-2Nb_2O_5, $MgTiO_3$/$CaTiO_3$ and $ZnNb_2O_6$/$SrTiO_3$ stacked ceramics [14-16]. As organic glues would be aged during operating, it would induce structural and functional failures to the devices. Probably because it is not easy to enable good sintering compatibility of different ceramic layers, few works have been reported upon cofired multilayer architectures (all-ceramic materials) in MWDCs. Especially for the materials that have opposite τ_f and cannot coexist well, such cofired multilayer ceramic architectures can offer profound opportunity to realize good temperature stability by stacking and cofiring them appropriately. Meanwhile, the stacking scheme behaves a very important role in the microwave dielectric performance of cofired multilayer ceramics [15,17]. In our previous work, $Zn_{1.01}Nb_2O_6$/TiO_2/$Zn_{1.01}Nb_2O_6$ was demonstrated to be the optimal stacking scheme among the available cofired multilayer ceramic architectures, which includes $Zn_{1.01}Nb_2O_6$/TiO_2 and TiO_2/$Zn_{1.01}Nb_2O_6$/TiO_2 [17]. When stacked with a small amount of TiO_2 (1.84 vol%), a superior microwave dielectric characteristic of ε_r ~ 26.8, $Q\times f$ ~ 99 500 GHz and near-zero τ_f could be

achieved in the cofired $Zn_{1.01}Nb_2O_6/TiO_2/Zn_{1.01}Nb_2O_6$ trilayer ceramics. As is known, $MgTiO_3$ has a high $Q\times f$ value ~ 160 000 GHz accompanied with ε_r ~ 17 and τ_f ~ −50 ppm/°C [9], and theoretically TiO_2 would also be an ideal temperature compensator for $MgTiO_3$. And yet, there are several compounds like Mg_2TiO_4 and $MgTi_2O_5$ existing in the MgO-TiO_2 binary system, thus it does not seem easy to adjust the τ_f of $MgTiO_3$ using TiO_2 additions through conventional solid-state mixed route [2,9]. Motivated by the design of cofired trilayer ceramic architectures in the $Zn_{1.01}Nb_2O_6$-TiO_2 system, herein an ultralow-loss ($Q\times f$ ~ 169 900 GHz) and temperature-stable microwave dielectric was achieved in the typical $MgTiO_3$-TiO_2 system with the stacking scheme of $MgTiO_3/TiO_2/MgTiO_3$. The effects of TiO_2 content on the microwave dielectric properties of the cofired ceramic architectures were studied.

2. Experimental Procedure

The layered $MgTiO_3/TiO_2/MgTiO_3$ ceramics were prepared through cofiring process based on our previous work [17], using high-purity MgO (99.99%, aladdin) and TiO_2 (99.99%, aladdin) powders as the raw materials. $MgTiO_3$ powders were prepared solely. Firstly, MgO and TiO_2 powders were weighed and ball-milled in ethanol for 4 h. Then, the slurry was dried and calcined at 1100 °C for 4 h. Generally, $MgTiO_3$ ceramic reveals a narrow sintering window and its densification temperature is approximately 1350 °C [2]. In our previous work, TiO_2 ceramics with relatively high densification (> 97%) could be obtained over a wide sintering temperature range of 1250~1500 °C, using the same TiO_2 powder as the raw materials and without any calcination involved [18]. In that case, the TiO_2 samples sintered at 1350°C have a size of ~ 8.24 mm in

diameter (its original size is 10 mm), which is much smaller than that of matrix $MgTiO_3$ ceramics (with ~ 8.72 mm in diameter). For a better sintering compatibility (densification temperature and shrinkage) with the matrix $MgTiO_3$ ceramics, the TiO_2 powders were pre-treated at 1100 °C for 4 h, followed by ball-milled and dried process before use. After the pre-treatment, a much closer size (with ~ 8.64 mm in diameter) to that of $MgTiO_3$ can be obtained for the TiO_2 samples. Before the layered process, the as-prepared $MgTiO_3$ and TiO_2 powders were ground with 5 wt% organic binders (PVA). After that, according to the stacking scheme of $MgTiO_3/TiO_2/MgTiO_3$, each layer was pre-pressed, stacked and finally pressed into cylinders (10 mm in diameter) with different volume fractions of TiO_2 (0.86~1.71 vol%). The total weight of $MgTiO_3$ in each cylinder was kept the same. After isostatically pressed at 200 MPa for 90 s, the samples were sintered at 1350 °C for 4 h in air at a heating rate of 5 °C /min.

The crystal phases of the as-prepared specimens were employed by X-ray diffraction (XRD, D8 Advance, Bruker, Karlsruhe, Germany). The microstructures of the samples were characterized by scanning electron microscopy (SEM, MERLIN VP Compact, Carl Zeiss, Jena, Germany). The microwave dielectric properties (ε_r, $Q\times f$ value and τ_f) were employed by a network analyzer (HP8720ES, Hewlett-Packard, Santa, Rosa, CA) accompanied with a temperature chamber (MC-811T, Espec, Osaka, Japan). The τ_f values were measured from room temperature (25 °C) to 80 °C.

3. Results and Discussion

Figure 1 depicts the XRD patterns of as-prepared $MgTiO_3$ and TiO_2 samples. As for the

reflection peaks of $MgTiO_3$ powders calcined at 1100 °C (Fig. 1a), the rhombohedral structure of $MgTiO_3$ (JCPDS 06-0494) is observed as the main phase, while $MgTi_2O_5$ (JCPDS 20-0694) and TiO_2 (Rutile, JCPDS 21-1276) as the secondary phases. After sintered at 1350 °C, the $MgTiO_3$ ceramics are identified as a pure rhombohedral phase displayed in Fig. 1b. For the TiO_2 calcined powders (1100 °C for 4 h) and sintered ceramics (1350 °C for 4 h) shown in Figs. 1c-d, all the reflection peaks are indexed to pure rutile phase (JCPDS 21-1276).

Figure 2 shows the cross-section micrographs of the $MgTiO_3/TiO_2/MgTiO_3$ layered ceramic architectures cofired at 1350 °C for 4 h, and the insets show the EDS results of the selected spots and line. The left side of the SEM image with small grains (where the Mg/Ti ratio is approximately 1:1, spot A) pertains to $MgTiO_3$ layer, while the right side with larger grains (without obvious detection of Mg element, spot D) belongs to TiO_2 layer. To be noted that the spots B and C along with the $MgTiO_3/TiO_2$ interface show a distinctive Mg/Ti ratio from that of spots A ($MgTiO_3$) and D (TiO_2). They almost exhibit a Mg/Ti ratio of 1:2, which is similar to that of $MgTi_2O_5$ compound. As is known, there are several compounds existing in the MgO-TiO_2 binary system, and $MgTi_2O_5$ usually appears as a secondary phase in $MgTiO_3$-based ceramics. During a high temperature sintering process, the chemical reactions between $MgTiO_3$ and TiO_2 would take place along with the $MgTiO_3/TiO_2$ interfaces, resulting in an intermediate layer as indicated by the red dotted lines. Such intermediate layer is approximately 15 μm in width, and importantly it could act as the "in-situ" glues to bond each layer during the sintering process [17]. For many reported MWDCs with stacking architectures, such as $MgTiO_3/CaTiO_3$ and $ZnNb_2O_6/SrTiO_3$, the layers were glued by organic binders [14-16]. However, it is inevitable for the aging of organic glues during operating, which could give rise to structural and functional

failures to the devices. In the case of cofired $MgTiO_3/TiO_2/MgTiO_3$ trilayer ceramics, by contrast, this all-ceramic architecture could enable good reliability against operating, which is beneficial for practical applications.

The microwave dielectric properties of cofired $MgTiO_3/TiO_2/MgTiO_3$ trilayer ceramics are depicted in Fig. 3. For a clear illustration, the theoretical microwave dielectric properties (ε_r and τ_f) of $MgTiO_3$-TiO_2 composite ceramics (in case $MgTiO_3$ and TiO_2 could coexist with each other) prepared via conventional solid-state mixed route are calculated by the empirical logarithmic rule as follows:

$$\log\varepsilon_r = v_1\log\varepsilon_1 + v_2\log\varepsilon_2 \quad (1)$$

$$\tau_f = v_1\tau_{f_1} + v_2\tau_{f_2} \quad (2)$$

where ε_1 and ε_2, τ_{f1} and τ_{f2}, and v_1 and v_2 are the dielectric constants, τ_f values and volume fractions of material 1 and material 2, respectively [3,19]. The calculated results are also given along with Fig. 3. Pure $MgTiO_3$ ceramic with simple configuration was prepared as comparison, which has ε_r ~ 16.69 and $Q\times f$ ~ 249 000 GHz. As the TiO_2 volume fraction increases from 0.86 vol% to 1.71 vol%, the measured resonant frequency (f_0) linearly decreases from 10.675 GHz to 10.172 GHz, while an increase from 17.17 to 18.75 for ε_r, a decrease from 197 300 GHz to 161 200 GHz for $Q\times f$ values, and a variation of −29 ppm/°C to +6 ppm/°C for τ_f. Meanwhile, the calculated ε_r and τ_f of $MgTiO_3$-TiO_2 composite ceramics are almost lower than their corresponding values of cofired trilayer ceramics. In particular, with TiO_2 volume fraction increasing from 0.86 vol% to 1.71 vol%, the τ_f values do not show much change of −46 ~ −41 ppm/°C. Based on the calculation results, the τ_f value could be tuned to near zero when the volume fraction of TiO_2 is approximately 10 vol%, in which ε_r would be ~ 20. By contrast, the cofired trilayer ceramic architectures have a

remarkable role in tuning the microwave dielectric performance of $MgTiO_3$-TiO_2 system materials. As is readily seen, with stacking a small amount of TiO_2 (1.63 vol%), the τ_f can be effectively tuned to ~ −1 ppm/°C, and the samples can possess an ultrahigh $Q\times f$ value ~ 169 900 GHz and a ε_r ~ 18.38 as well. As is known, $MgTiO_3$ and TiO_2 ceramics cannot coexist using the conventional solid-state mixed method [2,9]. However, in the case of cofired $MgTiO_3/TiO_2/MgTiO_3$ trilayer ceramics, the chemical reactions between $MgTiO_3$ and TiO_2 can be limited within a very narrow area (~15 μm, Fig. 2). Although the new phase ($MgTi_2O_5$) within the interfaces has a lower $Q\times f$ value (47 000 GHz) than that of $MgTiO_3$ (~160 000 GHz) [9,20], the effect of $MgTi_2O_5$ on the microwave dielectric properties of the layered architectures is also very limited. Thus, it is beneficial for the cofired $MgTiO_3/TiO_2/MgTiO_3$ trilayer ceramic architectures to achieve high $Q\times f$ values and near-zero τ_f simultaneously.

Figure 4 summarizes the microwave dielectric properties ($Q\times f$ and τ_f values) of typical MgO-TiO_2 system MWDCs, and some of them are listed in Table 1 [2,8,9,10,20-25]. It is obvious that the current cofired $MgTiO_3/TiO_2/MgTiO_3$ trilayer ceramics exhibit superior microwave dielectric properties than the available MgO-TiO_2 system materials. The findings indicate that the cofired multilayer ceramic architecture can be a useful and complementary way to conventional solid-state mixed process, which can therefore be beneficial for developing high-performance DRs and multilayers for 5G wireless communication applications.

4. Conclusions

An ultrahigh-Q and temperature-stable microwave dielectric was designed in the typical $MgTiO_3$-TiO_2 system via a cofired trilayer ceramic architecture showing as $MgTiO_3/TiO_2/MgTiO_3$.

With such layered ceramic architecture, the effects of the chemical reactions between $MgTiO_3$ and TiO_2 can be effectively limited to a narrow region (~15 μm in width) within the $MgTiO_3/TiO_2$ interfaces. Meanwhile, this intermediate region could act as the "glues" to bond each layer well. Excellent characteristics of ε_r ~ 18.38, $Q\times f$ value ~ 169 900 GHz and τ_f ~ −1 ppm/°C were achieved for the $MgTiO_3/TiO_2/MgTiO_3$ ceramic architectures stacked with 1.63 vol% TiO_2. The current findings could gain new strategies to develop high-performance dielectric resonators and multilayers for 5G wireless communication applications.

Acknowledgements

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Figure Captions

Figure 1 XRD patterns of as-prepared samples: (a) $MgTiO_3$ powders calcined at 1100 °C for 4 h, (b) $MgTiO_3$ ceramics sintered at 1350 °C for 4 h, (c) TiO_2 powders calcined at 1100 °C for 4 h, and (d) TiO_2 ceramics sintered at 1350 °C for 4 h.

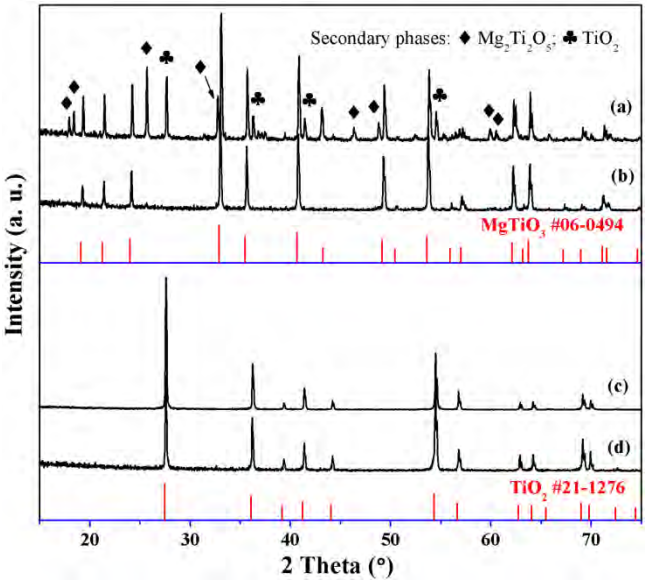
Figure 2 Cross-section micrographs of the interfaces between the two systems sintered at 1350 °C for 4 h, and insets show the EDS results of the selected line and spots.

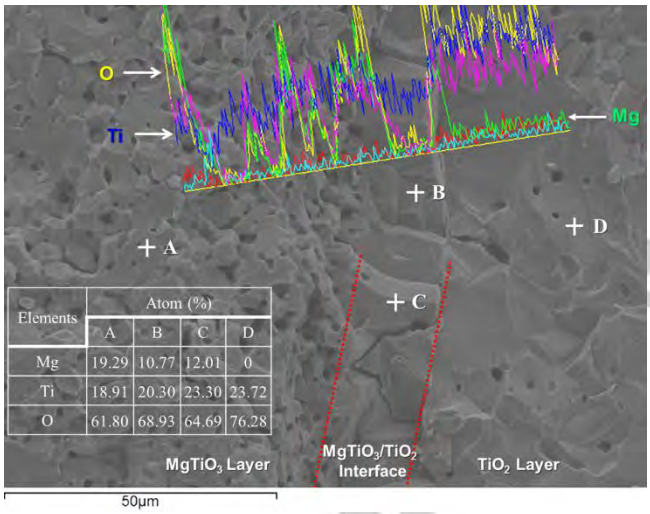
Figure 3 Microwave dielectric properties of $MgTiO_3/TiO_2/MgTiO_3$ trilayer ceramics cofired at 1350 °C for 4 h as a function of TiO_2 volume fraction: (a) resonant frequency (f_0), (b) ε_r, (c) $Q\times f$ value and (d) τ_f. The calculated ε_r and τ_f of $MgTiO_3$-TiO_2 composite ceramics are also given as comparison.

Figure 4 Summary of $Q\times f$ value versus τ_f plot for MgO-TiO_2 system MWDCs. The data are collected from literatures [2,8,9,10,20-25].

Table 1 Comparison of microwave dielectric properties of some typical MgO-TiO_2 system MWDCs [2,9,20-23].

Material systems (or method)	*T.S.* (°C)	ε_r	$Q\times f$ (GHz)	τ_f (ppm/°C)	Ref.
$MgTi_2O_5$	1500	17.4	47 000	-66	[20]
$MgTi_2O_5$+10 wt% LBS glass	950	19.3	6 800	-16	[20]
$MgTi_2O_4$	1450	14	150 000	-50	[21]
$0.93Mg_2TiO_4$–$0.07CaTiO_3$	1420	15	35 000	-2	[21]
$0.91Mg_2TiO_4$–$0.09(Ca_{0.8}Sr_{0.2})TiO_3$	1300	18.3	90500	0	[22]
$MgTiO_3$	1450	17	160 000	-50	[9]
$0.95MgTiO_3$–$0.05CaTiO_3$	1400	20	56 000	0	[9]
$0.96MgTiO_3$–$0.04SrTiO_3$	1300	20.96	135 000	0	[2]
$MgTiO_3$–$MgTi_2O_5$	1300	22.4	40 400	-9.6	[23]
$MgTiO_3/TiO_2/MgTiO_3$	1350	18.38	169 900	-1	This work





Elements	Atom (%)			
	A	B	C	D
Mg	19.29	10.77	12.01	0
Ti	18.91	20.30	23.30	23.72
O	61.80	68.93	64.69	76.28