Temperature-stable Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂ garnet-type 5G millimeter-wave dielectric ceramic resonator antenna

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ABSTRACT

Temperature stability is a crucial property of microwave electronic components, as well as a pivotal aspect of assessing the performance of microwave dielectric ceramics. In this article. the temperature coefficient of high-Qxf garnet-type Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂ microwave ceramic was regulated by doping with different mass ratios of TiO₂. Further, combined with the Kramers-Kronig (K-K) formula, the dielectric loss and theoretical permittivity of Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ ceramic are calculated by infrared reflection spectrum data, which coincide exactly with the experimental results. Importantly, a 5G millimeter-wave antenna was fabricated with Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ ceramic and tested at 25 °C and 85 °C, respectively. The center frequencies of measurement are 25.99 GHz at 25 °C and 26.12 GHz at 85 °C, and the frequency shift with temperature is rather low, showing excellent temperature stability. The simulated efficiency of 88.5% and gain of 6.05 dBi also indicate that the antenna has favorable radiation characteristics. The results show that the temperature-stable Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ ceramic antenna has broad prospect in 5G millimeter wave communication.

Keywords: Infrared reflectivity spectroscopy; Garnet; Millimeter-wave antenna

1. Introduction

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As the amount of data in the network continues to increase, the communication system needs to improve the transmission speed and carrying capacity, and the frequency band trend of mobile communication is also moving towards the high frequency band [1-3]. The high-frequency band of 5G mobile communication is in the millimeter wave region [4]. In the future, 6G communication frequency will rise to the terahertz frequency band. The continuous increase in frequency also puts forward higher requirements for ceramic materials [5-7]. Ceramic materials are an indispensable part of electronic components. Compared to metal materials, ceramic materials have the stable temperature coefficient and ultra-low dielectric loss. These advantages are especially evident in high-frequency communication systems [8-10].

Ceramic materials are widely used in electronic components, such as dielectric substrates, dielectric resonators, ceramic filters, etc. [11-15]. J.F. Ziircher [16] designed and tested microstrip patch antennas for GPS systems using ceramic material and RT/Duroid 6010 dielectric substrate, respectively, over a temperature range of -5 to +44 °C. The results show that temperature has a large influence on the resonance frequency of the RT/Duroid 6010 antenna, and the applicable temperature range is narrow, ranging from 15 to 25 °C. While with a ceramic substrate, the antenna's center frequency changes very little with temperature (about 0.2%) and matches well across all thermal ranges. And for dielectric resonators, G. Drossos *et al.* [17] designed a cylindrical dielectric resonator antenna and examined its performance at different temperatures (300 K and 77 K). The resonance frequency and return loss of the DRA vary with temperature. It is noticeable that temperature stability must also be regulated in addition to enhance the $Q \times f$ value of microwave dielectric ceramics.

Garnet-typed microwave ceramics have attracted more attention owing to their low ε_r , high $Q \times f$ value and adjustable τ_f [18-21]. Furthermore, when compared to other low dielectric constant systems, garnet has a stable crystal structure, a wide range of synthesis stability, and a simple preparation process, all of which contribute to its high application potential [22-23]. The dielectric constant of garnet-type microwave dielectric ceramics is generally around 10, making them low-dielectric ceramics that are ideal for low-latency 5G high-frequency communication [24]. In addition to dielectric constant, ceramic materials used in electronic components must have a high $Q \times f$ and a near-zero τ_f [25-27]. Zhou *et al.* [28] reported a very high $Q \times f$ of 234,936 GHz by adding excess Y and sintering in a vacuum of 1750 °C for 12 hours, without adjusting the τ_{f} , and with a high sintering temperature and long sintering time.

The authors' group studied Y₃MgAl₃SiO₁₂ garnet-type ceramics, which successfully reduced the sintering temperature to 1550 °C and the sintering time to 4 hours, while exhibiting excellent microwave dielectric properties: $\varepsilon_r = 10.1$, $Q \times f = 57,340$ GHz and $\tau_f = -32$ ppm/°C [29]. Further, the $Q \times f$ was increased by ~20% via the strategy of Dy³⁺ substitution in the Y³⁺ lattice site for Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂ microwave ceramic ($\varepsilon_r = 9.68$, $Q \times f = 68,866$ GHz, $\tau_f = -38.5$ ppm/°C). However, it still has a large negative value for its temperature coefficient, making its practical application challenging. In order to adjust τ_f of Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂ ceramic to near zero, a material with a positive temperature coefficient, such as TiO₂ ($\tau_f \sim +460$ ppm/°C) was added as compensation [30-31]. (1-*x*)Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂- *x*wt%TiO₂ (*x* = 0-9) microwave ceramics were therefore prepared, and the effects of various TiO₂ concentrations on bulk density, grain size and dielectric properties were studied. Moreover, a millimeter-wave antenna was fabricated based on temperature stable Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ ceramic and tested at 25 °C and 85 °C, respectively.

2. Experimental

2.1 Synthetic samples

 $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-x$ wt%TiO₂ (x = 0.9) ceramics were synthesized by solid-state reaction method. High-purity raw materials of Y₂O₃ (99.99%), Dy₂O₃ (99.99%), MgO (99.99%), Al₂O₃ (99.99%), SiO₂ (99.99%) were weighed on the basis of stoichiometric ratio and the obtained raw materials were ball-milled in solvent ethanol for 12 h. And the dried powders were calcined at 1575 °C for 4 h. The composite materials were ball milled in absolute ethanol for 12 h after adding different mass ratios of TiO₂. The mixed powders were calcined at 1300 °C for 4 hours. The calcined powders were reground and uniformly mixed with 5wt% PVA. The powders were pressed into cylindrical particles (thickness: 7-8mm, diameter: 12.7mm) and then sintered at 1450-1625 °C for 5 h.

2.2 Characterization

The phase compositions and crystal structure were identified by X-ray diffraction (Shimadzu, Kyoto, Japan) using Cu-K α radiation with a scanning angle of 10-80° and a step size of 0.02°. The microstructure was observed by the scanning electron microscope (Sigma 300, ZEISS). The Archimedes method was used to determine the bulk density. Microwave dielectric properties were measured in TE_{01δ} mode using the resonant cavity method. The Keysight (N5234B) vector network analyzer was used for evaluating the $Q \times f$ values and ε_r as well as the return loss (S_{11}) measurement. The τ_f value was obtained by the following formula:

$$\tau_f = \frac{f_{85} - f_{25}}{f_{25} \times (85 - 25)} \tag{1}$$

where f_{25} and f_{85} denote the resonance frequencies at 25 °C and 85°C, respectively.

2.3 Antenna design and fabrication

The dielectric resonator antenna is simulated by HFSS, and the DRA size is related to $\frac{\lambda_0}{\sqrt{\varepsilon_r}}$, where λ_0 is the wavelength at resonant frequency and ε_r is the dielectric constant

of the DR. Thus for the same frequency, a high dielectric constant can reduce the size of DR [32].

The design formulas of typical rectangular dielectric resonant antenna by means of numerical calculation and curve fitting are as follows [33-34]:

$$f_{GHz} = \frac{15F}{w_{cm}\pi\sqrt{\varepsilon_r}}$$
(2)

$$F = a_0 + a_1(\frac{w}{b}) + a_2(\frac{w}{b})^2$$
(3)

$$a_0 = 2.57 - 0.8(\frac{d}{b}) + 0.42(\frac{d}{b})^2 - 0.05(\frac{d}{b})^3$$
(4)

$$a_1 = 2.71 (\frac{d}{b})^{-0.282} \tag{5}$$

$$a_2 = 0.16$$
 (6)

where w_{cm} is expressed in cm and the resonant frequency will be in GHz, $w \times d \times b/2$ is

the dimension of the DRA.

The following formulas can be used as a starting point for rectangular slots:

$$l_s = \frac{0.4\lambda_0}{\sqrt{\varepsilon_e}} \tag{7}$$

$$\varepsilon_e = \frac{\varepsilon_r + \varepsilon_s}{2} \tag{8}$$

$$w_s = 0.2l_s \tag{9}$$

where l_s and w_s are the slot length and width, respectively, ε_r and ε_s are the dielectric constants of the DRA and substrate, respectively.

The stub extension *s* is selected so that its reactance cancels out that of the slot aperture. It is generally initially chosen to be:

$$s = \frac{\lambda_g}{4} \tag{10}$$

where λ_g is the guide wave in the substrate.

The proposed antenna consisted primarily of a dielectric substrate, a rectangular dielectric resonator fabricated by $Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$ -9wt%TiO₂, and a SMA connector that was excited by aperture coupling and exhibited excellent radiation characters. The rectangular dielectric resonator of the antenna was obtained by grinding the cylindrical sample into a rectangle. And an adhesive tape on the grounding plate is used to fix the dielectric resonator. The Keysight (N5234B) supports a frequency range of 10 MHz to 43.5 GHz, and the SMA connector supports a frequency range of DC - 40 GHz. And the antenna was tested at different temperature (25 °C and 85 °C). The antenna was heated on a heating table, and the temperature of the antenna was measured using an infrared thermometer (-32 °C~380 °C) to ensure the temperature reached 85 °C.

3. Results and discussion

The bulk densities of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-x$ wt%TiO₂ (x = 0-9) ceramics sintered at 1450 °C-1625 °C are plotted in Fig. 1(a). The bulk densities of composite ceramics containing various mass fractions of TiO₂ increase markedly and then decrease as sintering temperature rising. Furthermore, the maximum bulk density of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-x$ wt%TiO₂ (x = 0.9) ceramics gradually decreases from 4.46 g/cm³ to 4.3 g/cm³ as the TiO₂ mass fraction increases. This is because density of TiO₂ (4.23 g/cm³) is lower than that of Y_{2.95}Dy_{0.05}MgAl_3SiO_{12} (4.46 g/cm³) [35]. The optimal sintering temperature decreases as the TiO₂ content increases, indicating that TiO₂ is beneficial in reducing the sintering temperature. Fig. 1 (b) shows the relative densities of TiO₂ doped with different mass fractions at the optimum sintering temperature. With the increase of TiO₂ doping content, the relative density decreases from 98.28% (x=0) to 94.55% (x=9), which is consistent with the change trend of $O \times f$ value.



Fig. 1. The bulk densities of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-xwt\%TiO_2$ (*x*=0-9) composite ceramics varies with sintering temperature. (b) Relative densities of TiO₂ doped with different mass fractions at the optimum sintering temperature.

Fig. 2 illustrates the XRD patterns of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-x$ wt%TiO₂ (x = 0.9) ceramics at optimal sintering temperature. When TiO₂ is not added (x=0), all diffraction peaks are indexed to $Y_3Al_5O_{12}$ (PDF#82-0575) garnet. As the mass fraction of TiO₂ increases, the main diffraction peaks correspond to the $Y_3Al_5O_{12}$ phase, while the others correspond to two phases of TiO₂ (PDF#73-1764) and $Y_2Ti_2O_7$ (PDF#85-1570). The appearance of the $Y_2Ti_2O_7$ phase is due to the limited solid solubility of TiO₂ in $Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$. When x = 3, the phases of TiO₂ and $Y_2Ti_2O_7$ are not obvious, and a portion of TiO₂ may be solid-dissolved into $Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$ crystal. With increasing TiO₂ concentration, the phases of TiO₂ and $Y_2Ti_2O_7$ gradually become more stable. When the TiO₂ concentration exceeds the limiting value, the secondary phase $Y_2Ti_2O_7$ forms, as confirmed in the published literature [36-38].



Fig. 2. XRD of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-x$ wt%TiO₂ (x = 0-9) ceramics.

The SEM micrographs of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-xwt\%TiO_2(x=0-9)$ ceramics are demonstrated in Fig. 3(a-e). The average grain size and grain distribution of the composite ceramic samples, as measured by Image J software, are shown in the insets. From the SEM images of all samples (Fig. 3 (a-e)), the surface grain boundaries of the composite ceramic samples are relatively clear, the grain size distribution is uniform, and there are no obvious pores, indicating that all composite ceramics have relatively high densities. Fig. 3(f) shows the trend of the average grain size of composite ceramics varies with TiO₂ content. As the mass percentage of TiO₂ increases, the average grain size falls off from 6.12 μ m (*x*=0) to 4.20 μ m (*x*=9), indicating that the increase of TiO₂ content inhibits the growth of Y_{2.95}Dy_{0.05}MgAl_3SiO₁₂ ceramic grains.



Fig. 3. SEM images of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}-x$ wt%TiO₂ (x=0-9) ceramics: (a) x = 0; (b) x = 3; (c) x = 5; (d) x = 7; (e) x = 9. (f) The average grain size of composite ceramics.

Fig. 4 plots the microwave dielectric properties of $(1-x)Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$ xwt%TiO₂ (x = 0-9) ceramics. The ε_r increases gradually from 9.68 (x = 0) to 12.06 (x = 9), as shown by the changing trend of the black curve in Fig. 4. This is because the TiO₂ ($\varepsilon_r \sim 105$) possesses a higher dielectric constant than Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂ ceramic ($\varepsilon_r \sim 9.68$) [39]. The red curve represents the trend of change in the $Q \times f$ value of composite ceramics with various mass fractions of TiO₂. And the $Q \times f$ value drops sharply from 68,866 GHz to 28,604 GHz because the $Q \times f$ value of TiO₂ (~46,000 GHz) is lower than that of Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂ (~68,866 GHz), and the addition of TiO₂ introduces the secondary phase Y₂Ti₂O₇, whose $Q \times f$ value is only 9,000 GHz, which is much smaller than TiO₂ (~ 46,000 GHz) and Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂ (~68,866 GHz) [36,40]. In addition, the grain boundaries among Y_{2.95}Dy_{0.05}MgAl₃ SiO₁₂, TiO₂ and Y₂Ti₂O₇ increase as the mass fraction of TiO₂ increases, resulting in greater lattice distortion between different grain boundaries, which is also one of the reasons for the sharp decline in $Q \times f$ value. And the blue curve is the variation trend of the temperature coefficient (τ_f) of the composite ceramics. Since TiO₂ has a high positive temperature coefficient (τ_{f} + 460 ppm/°C), increasing the mass fraction of TiO₂ improves the temperature coefficient of the composite ceramics effectively [41]. When x = 9, the temperature coefficient is close to zero and the specific τ_f value is -3.4ppm/°C.



Fig. 4. Microwave dielectric properties of (1-x) Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-x wt%TiO₂ (x= 0-9) ceramics.

For purpose of further analyzing the association between the inherent microwave dielectric properties of $Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$ -9wt%TiO₂ ceramic and lattice vibration, the infrared spectral data of the ceramic was analyzed based on classical harmonic oscillator model:

$$\varepsilon^*(\omega) = \varepsilon_{\infty} + \sum_{j=1}^n \frac{(z_j e)^2 / m_j V_j \varepsilon_0}{\omega_{Tj}^2 - \omega^2 - j\omega\gamma}$$
(11)

The z_j , m_j , V_j , γ_j , ω_{Tj} , and n in the above equation have been described at great length in the literature[42]. The association between permittivity and complex reflectance $R(\omega)$ can be represented as:

$$R(\omega) = \left| \frac{1 - \sqrt{\varepsilon^*(\omega)}}{1 + \sqrt{\varepsilon^*(\omega)}} \right|^2$$
(12)

According to Equations (11) and (12), the infrared reflectance spectrum of $Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$ -9wt%TiO₂ ceramic can be well fitted. In the microwave frequency region ($\omega \ll \omega_{Pj}$), the imaginary and real parts of complex permittivity ($\varepsilon^*(\omega)$) can be expressed as Equations (13) and (14) [43].

$$\varepsilon''(\omega) = \omega \sum_{j=1}^{n} \frac{\partial \varepsilon_{j} \gamma_{j}}{\omega_{T_{j}}^{2}}$$
(13)

$$\varepsilon'(\omega) = \varepsilon(\infty) + \sum_{j=1}^{n} \frac{\omega_{P_j}^2}{\omega_{T_j}^2} = \varepsilon(\infty) + \sum_{j=1}^{n} \partial \varepsilon_j$$
(14)

As shown in Fig. 5(a), the infrared reflectance spectrum can be well fitted with twelve Lorentzian modes, and the results are shown in Table 1. And the theoretical ε_r (~12.71) at 9.53 GHz is close to the measured value (~12.02). The calculated $Q \times f$ value of 33,321 GHz (f = 9.53 GHz, $Q = 1/\tan \delta$, $\tan \delta = 2.86 \times 10^{-4}$) exceeds the measured value (28,604 GHz). The difference between measured and calculated values owing to the contribution of secondary phase, grain boundary and grain size to $Q \times f$ value.



Fig. 5. (a) Fitted and experimental infrared reflection spectrum and (b-c) fitted complex dielectric spectrum of Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ ceramic.

Table 1

Phonon parameters obtained from the fitting of the infrared reflectivity spectra of $Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$ -9wt%TiO₂ ceramic.

Mode	ω_{pj}	ω _{oj}	$\gamma_{\rm j}$	$ riangle \epsilon_{j}$	$tan\delta_j \times 10^{-4}$	
1	31.823	90.865	12.864	0.123	0.142	
2	133.58	163.45	15.808	0.668	0.0967	
3	190.1	213.17	29.494	0.795	0.138	
4	357.66	279.55	102.93	1.64	0.368	
5	196.58	317.29	35.596	0.384	0.112	
6	400.31	431.15	64.337	0.862	0.149	
7	182.2	472.57	51.073	0.149	0.108	
8	147	514.56	34.747	0.0816	0.0675	
9	112.39	575.51	29.599	0.0381	0.0514	
10	438.37	684.41	162.03	0.41	0.237	
11	220.39	796.23	99.892	0.0766	0.125	
12	255.29	931.76	173.61	0.0751	0.186	

Dielectric resonators are one of the most important applications of microwave dielectric ceramics [44-46]. Following the publication of dielectric resonator antennas by Long et al. [47-48], the design of dielectric resonators as radiating antennas has gradually gained wide attention. For illustrating the possible application and goodness of temperature stable Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ ceramic, a temperature-stable 5G millimeter-wave dielectric resonator antenna was fabricated.

Fig. 6(a) and (b) depict the 3D structure diagram of the designed antenna. The antenna is fed using aperture coupling, with a rectangular dielectric resonator positioned directly above the aperture. The microstrip line is located at the bottom of the FR4 substrate, where two circular holes (r = 1.05 mm) facilitate the installation of SMA connector. The sizes of the DR, FR4 substrate and slot are $a \times a \times h$, $x \times x \times h_0$ and l_s $\times w_s$ (unit: mm), respectively. l_m is the stub extension and w_f denotes the width of the microstrip line. The specific parameter values are listed in Table 2. Fig. 6(c) and (d) are the upper surface and the lower surface of actual antenna, respectively. Fig. 6(e) shows that the measured and simulated S_{11} show good consistency. The center frequencies of measurement at 25 °C and 85 °C are 25.99 GHz and 26.12 GHz, respectively. The results prove the frequency shift is quite low, about 0.5%, showing a stable temperature property of the ceramic antenna. The bandwidth (S_{11} < -10dB) of measurement at 25°C and 85 °C are 1.190 GHz and 1.140 GHz, respectively, which reflect the broadband characteristic of dielectric resonator antenna. Fig. 6 (f) shows the simulated impedance at 26.10 GHz. The impedance value of $(50.29+1.14j) \Omega$ is very close to 50 Ω , indicating that impedance matching is excellent, which can also be confirmed by the values of S_{II} in Fig. 6(e) are less than -30 dB. The simulated efficiency and gain in Fig. 6(g) are 88.5% and 6.05 dBi, respectively. Fig. 6(h) and (i) show the patterns of the cross-polarization and co-polarization of the E-plane and H-plane. In the boresight direction ($\theta=0^{\circ}$), the co-polarization field is over 20 dB stronger than cross-polarization field. Fig. 6 (j) and (k) are the three dimensional far-field radiation patterns at 26.10 GHz, which exhibit favorable radiation characteristics. According to the above results, the temperaturestable Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ ceramic antenna has a high potential application in 5G millimeter wave band.



Fig. 6. (a) 3D structure drawing and (b) specific dimensions of the proposed antenna. (c) The upper surface and (d) the lower surface of actual antenna. (e) Simulated and measured return loss (S_{11}) at 25 °C and 85 °C. (f) Simulated impedance of antenna. (g) Simulated efficiency and gain with various frequency. Cross-polarized and co-polarized of simulated (h) E-plane and (i) H-plane, (j) top and (k) bottom of simulated 3D radiation pattern at 26.10 GHz.

Table 2

The optimum parameters of the dielectric resonator antenna.

Parameters	а	h	x	h_0	ls	W_S	l_m	Wf	r
Values(mm)	4.3	2.4	20	0.6	2.1	1.6	0.2	0.6	1.05

4. Conclusions

In this work, various mass percentages of TiO_2 were added to form composite ceramics to regulate the temperature coefficient of the $Y_{2.95}Dy_{0.05}MgAl_3SiO_{12}$ ceramic. The XRD patterns revealed that the main crystalline phase remained $Y_3Al_5O_{12}$ after the addition of TiO₂, while Y₂Ti₂O₇ and TiO₂ appeared as secondary phases. Increasing the amount of TiO₂ decreased the *Q×f* value while increasing the ε_r and τ_f . Temperature stable composite ceramics are achieved for x = 9 wt% with $\varepsilon_r = 12.06$, $Q \times f = 28,604$ GHz (f = 9.53 GHz), $\tau_f = -3.4$ ppm/°C. The 5G millimeter-wave dielectric resonator antenna was fabricated based on the Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂- 9wt% TiO₂ ceramic and tested at 25 °C and 85 °C. The center frequencies of measurement at 25 °C and 85 °C are 25.99 GHz and 26.15 GHz, respectively, with the quite lower frequency shift of about 0.6%. The results suggest that the dielectric resonator antenna fabricated by temperature-stable Y_{2.95}Dy_{0.05}MgAl₃SiO₁₂-9wt%TiO₂ceramic has excellent temperature stability in millimeter wave band.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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