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Microwave dielectric properties of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ and $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2-0.8) ceramics

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Abstract

$SrLa[Ga_{1-x}(R_{0.5}Ti_{0.5})_x]O_4$ (R = Mg, Zn) ceramics were prepared by a standard solid state sintering method. The single-phase ceramics with K_2NiF_4-type layered perovskite structure and $I4$/mmm space group were obtained, indicating that $SrLa(R_{0.5}Ti_{0.5})$ and $SrLaGaO_4$ can form the unlimited solid solutions. With increasing x for R = Mg and Zn, ε_r increases monotonously, the Qf value first increases and then decreases, while τ_f increases from a negative to a positive value. The optimized microwave dielectric properties were obtained as following: ε_r = 23.3, Qf = 89 400 GHz, τ_f = −0.8 ppm/°C for $SrLa[Ga_{0.6}(Mg_{0.5}Ti_{0.5})_{0.4}]O_4$ and ε_r = 23.3, Qf = 76 200 GHz, τ_f = 0.2 ppm/°C for $SrLa[Ga_{0.7}(Zn_{0.5}Ti_{0.5})_{0.3}]O_4$, indicating that the present solid solution ceramics are the promising candidates as microwave resonator materials for the telecommunication applications.

KEYWORDS

K_2NiF_4-type structure, microwave dielectric properties

1 | INTRODUCTION

Microwave dielectric ceramics have attracted continuous attention in the past decades due to the rapid development of telecommunication technology, especially the mobile communication from 1G to 5G.[1,2] Served as the key resonator components in the microwave circuits,[3–6] the microwave dielectric ceramics are required to possess a high dielectric constant (ε_r), a high Qf value, and a near-zero temperature coefficient of resonant frequency (τ_f). The near-zero τ_f is particularly important considering the complex working conditions.

In the recent years, $MLnAlO_4$-based microwave dielectric ceramics (M = Sr and Ca, Ln = La, Nd, Sm and Y) with K_2NiF_4-type layered perovskite structure have been investigated systematically.[7–11] The good microwave dielectric properties with ε_r = 18-22, Qf = 40 000-99 400 GHz and near-zero τ_f have been obtained in a series of $MLnAlO_4$-based ceramics.[7–15] Al^{3+} in $MLnAlO_4$ can be replaced by the trivalent cations with higher ionic polarizabilities such as Ga^{3+} and $(R_{0.5}Ti_{0.5})^{3+}$ (R = Mg, Zn), and higher ε_r can be obtained in this way.[16] For example, the following microwave dielectric properties have been reported in $SrLaBO_4$ (B = Ga, $(Mg_{0.5}Ti_{0.5})$, $(Zn_{0.5}Ti_{0.5})$) ceramics: ε_r = 20.3, Qf = 16 200 GHz, τ_f = −33.5 ppm/°C for $SrLaGaO_4$,[17] ε_r = 25.5, Qf = 72 000 GHz, τ_f = +29 ppm/°C for $SrLa(Mg_{0.5}Ti_{0.5})O_4$, and ε_r = 29.4, Qf = 34 000 GHz, and τ_f = +38 ppm/°C for $SrLa(Zn_{0.5}Ti_{0.5})O_4$. Considering that the τ_f value of $SrLaGaO_4$ is negative while those of $SrLa(R_{0.5}Ti_{0.5})O_4$ are positive, the near-zero τ_f is expected in $SrLaGaO_4$-$SrLa(R_{0.5}Ti_{0.5})O_4$ solid solutions. On the other hand, Ren et al predicated that $SrLaAlO_4$, $SrLaGaO_4$, and $SrLa(R_{0.5}Ti_{0.5})O_4$ can form the unlimited solid solutions with each other according to the discussion on the stability of K_2NiF_4-type structure in $MLnBO_4$ (M = Ca, Sr, Ba; Ln = Y, Sm, Nd, La; B = Al, Ga, $(Mg_{0.5}Ti_{0.5})$, $(Zn_{0.5}Ti_{0.5})$)

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compounds in relation to the tolerance factor of perovskite layer and the radius ratio of M^{2+} and Ln^{3+}.[16] The prediction has been proven in $SrLa[Al_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ and $SrLa[Al_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics.[18,19] In the present work, therefore, $SrLa[Ga_{1-x}(R_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2-0.8) solid solution ceramics were prepared, and the microwave dielectric properties were investigated.

2 | EXPERIMENTAL PROCEDURE

$SrLa[Ga_{1-x}(R_{0.5}Ti_{0.5})_x]O_4$ solid solution ceramics were prepared by a standard solid state sintering method. High-purity raw powders of $SrCO_3$ (99.95%), La_2O_3 (99.99%), Ga_2O_3 (99.9%), MgO (99.9%), ZnO (99.95%), and TiO_2 (99.9%) were weighed according to the mole ratio. The powders were ball-milled using zirconia balls in ethanol for 24 hours and then dried. In order to obtain the single-phase powders, the mixtures were calcined in air for 3 hours at 1300°C for $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ and 1200°C for $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$, respectively. After ballmilling and drying again, the powders were mixed with 7 wt% PVA solution, and then pressed into compacts with 12 mm in diameter and 2-6 mm in height under a uniaxial pressure of 100 MPa. The compacts were sintered at 1275°C-1550°C in air for 3 hours to obtain the dense ceramics.

The bulk density of the ceramics was measured by Archimedes' method. Powder X-ray diffraction (XRD) with Cu Kα radiation (Rigaku D/max 2550/PC, Rigaku Co.) was used to identify the phase constitution and crystal structure. The ceramics were polished and thermally etched at the temperatures 50°C lower than the sintering temperatures, and the microstructures on the thermally etched surfaces were observed by scanning electron microscopy (S-4800, Hitachi). The cylindrical samples with about 10 mm in diameter and 5 mm in height were used to evaluate the microwave dielectric properties. A vector network analyzer (E8363B Agilent Technologies Inc) was used to measure ε_r and τ_f between 20°C and 80°C by the Hakki-Coleman method,[20,21] and the Qf value by the resonant cavity method.[22,23]

3 | RESULTS AND DISCUSSION

3.1 | $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics

Figure 1 shows the relative density of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2, 0.4, 0.6, 0.8) ceramics as a function of sintering temperature. The relative density first increases, and then decreases for all the compositions. The highest relative density is 97%–98%, and the sintering temperature for the highest density increases with x. The high relative density is confirmed by the dense microstructures on the thermally etched surfaces of the ceramics with the highest densities, as shown in Figure 2. The grains become larger with increasing x, which may be attributed to the changing composition and increasing sintering temperature. Besides, the microstructure is homogenous for x = 0.2 and 0.6, while the inhomogenous microstructure with bright particles are observed for 0.4 and 0.8.

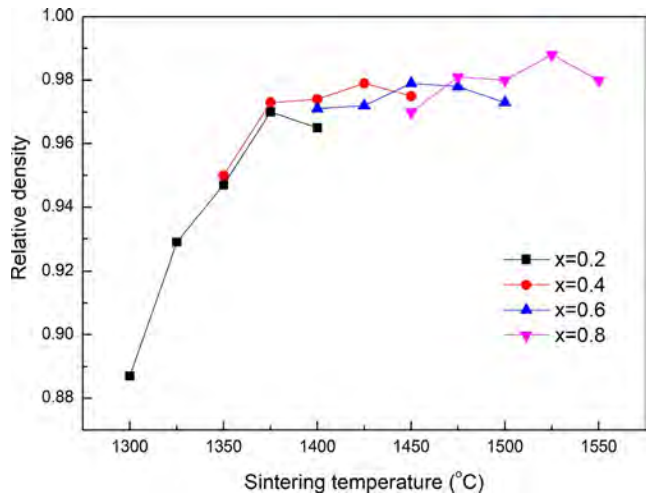

FIGURE 1 Relative density of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2, 0.4, 0.6, 0.8) ceramics as a function of sintering temperature

The XRD patterns of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics sintered at the optimal temperatures are shown in Figure 3. All the diffraction peaks match the characteristic peaks of $SrLaGaO_4$ (JCPDS#80-1806), indicating the single phase with K_2NiF_4-type layered perovskite structure and I4/mmm space group for $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics. The XRD results confirm the prediction on the $SrLaGaO_4$-$SrLa(Mg_{0.5}Ti_{0.5})O_4$ unlimited solid solution by Ren et al according to the discussion on the stability of K_2NiF_4-type structure in $MLnBO_4$.[16] It should be noted that the inhomogenous microstructure with bright particles are observed from SEM images for x = 0.4 and 0.8, but no secondary phase is revealed by XRD, indicating that the inhomogenous microstructure is formed on the surface during thermal etching, but does not exist inside the ceramic body.

Figure 4 shows the microwave dielectric properties of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics as functions of sintering temperature. The dielectric constant first rises and then declines slightly for all the compositions. The variation is consistent with the relative density as a function of sintering temperature shown in Figure 1. When x = 0.2-0.6, the Qf value drops after peaking at a high value. However, the Qf value fluctuates mildly between 60 000 and 70 000 GHz with the sintering temperature for x = 0.8. For all the compositions, the sintering temperature for the highest Qf value is lower than that for the highest relative density, and this may be due to the oxygen vacancies, whose negative effect on the Qf value becomes significant with increasing the sintering

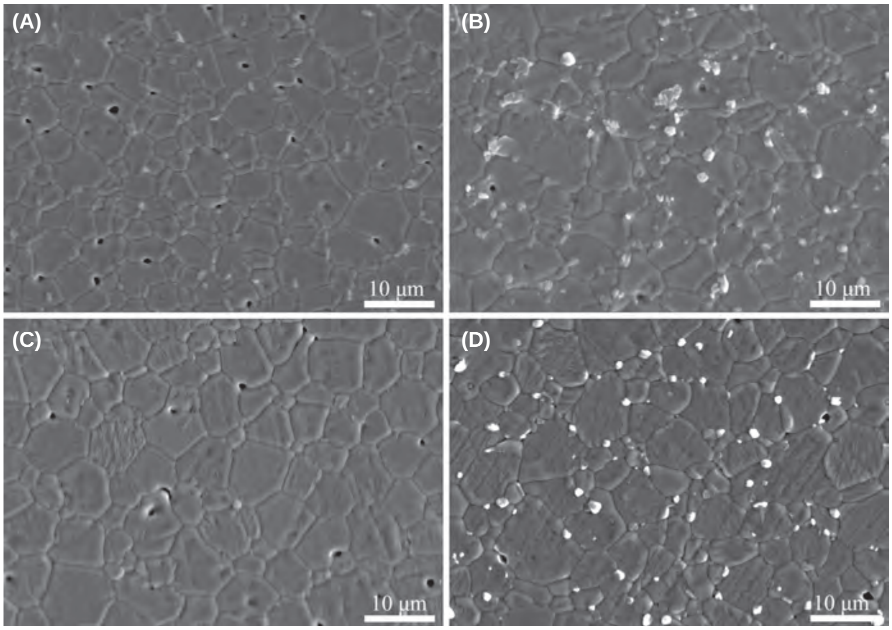

FIGURE 2 SEM images on thermally etched surfaces of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics with the highest densities: (A) $x = 0.2$ sintered at 1375°C, (B) $x = 0.4$ sintered at 1425°C, (C) $x = 0.6$ sintered at 1450°C and (D) $x = 0.8$ sintered at 1525°C

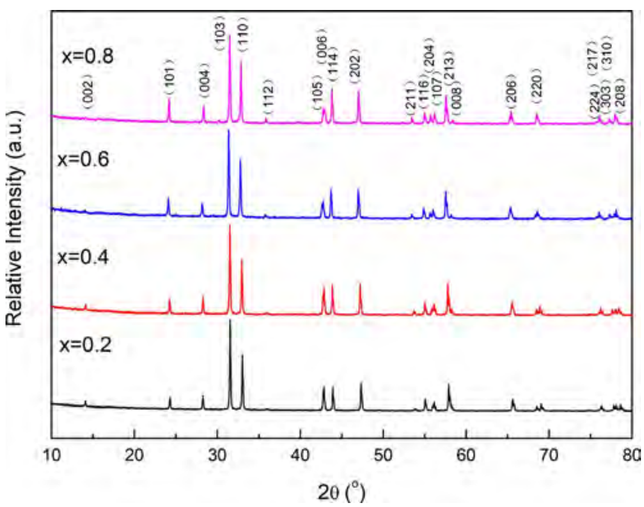

FIGURE 3 X-ray diffraction patterns of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ($x = 0.2, 0.4, 0.6, 0.8$) ceramics

temperature. Oxygen vacancies generally form in oxide ceramics during the high-temperature sintering, and more oxygen vacancies are expected to form at higher temperatures. Besides, the grains become larger for higher sintering temperatures, which make it more difficult for the oxygen in air to penetrate the grains and reduce the oxygen vacancies during the cooling process after sintering. As a result, the concentration of the residual oxygen vacancies increases with the sintering temperature, which is responsible for the lower *Qf* value for higher sintering temperatures. Furthermore, τ_f is not sensitive to the sintering temperature comparing with ε_r and *Qf*.

Figure 5 shows the optimal microwave dielectric properties of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics as functions of x. With x increasing from 0 to 1, the dielectric constant increases monotonically from 20.9 to 25.5, and the temperature coefficient of resonant frequency increases from −25.8 to 29 ppm/°C. Different from the monotonical increase of ε_r and τ_f with x, the *Qf* value first increases from 53 900 GHz ($x = 0$) to 92 800 GHz ($x = 0.6$), while it drops to 67 100 GHz for $x = 0.8$ and 72 500 GHz for $x = 1$. The *Qf* value is determined by the intrinsic and extrinsic factors. It is well known that the tolerance factor t of the perovskite layer can be used to evaluate the stability of K_2NiF_4-type structure,[24,25] which is defined as

$$t = \frac{\left[r\left(\mathrm{Sr}^{2+}\right) + r\left(\mathrm{La}^{3+}\right)\right]/2 + r\left(\mathrm{O}^{2-}\right)}{\sqrt{2}\left[r\left(\mathrm{B}^{3+}\right) + r\left(\mathrm{O}^{2-}\right)\right]}, \qquad (1)$$

for $SrLaBO_4$,[7] where r is the ionic radius. With x increasing from 0 to 1, t decreases nearly linearly from 0.932 to 0.917, indicating the decreasing structural stability and intrinsic *Qf* value. On the other hand, the $SrLaGaO_4$ single crystal was reported by Pajaczkowska et al[26] to possess a *Qf* value as high as 170 000 GHz. However, the *Qf* value of $SrLaGaO_4$ ceramic is 53 900 GHz in the present work, and it is only 16 200 GHz in a previous study.[17] The large difference indicates that the *Qf* value is sensitive to the extrinsic factors, which may also plays a role in the *Qf* variation with x in $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics. The good combination of microwave dielectric properties with $\varepsilon_r = 23.3$, $Qf = 89\ 400$ GHz and $\tau_f = -0.8$ ppm/°C are obtained for $SrLa[Ga_{0.6}(Mg_{0.5}Ti_{0.5})_{0.4}]O_4$ ceramics sintered at 1400°C. Besides, benefiting from the higher dielectric constant of $SrLaGaO_4$ ($\varepsilon_r = 20.9$) than $SrLaAlO_4$

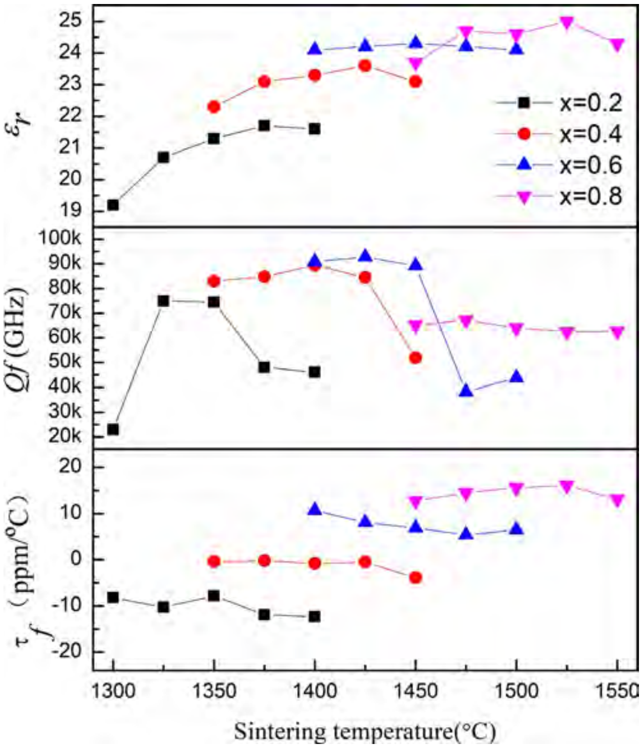

FIGURE 4 Microwave dielectric properties of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2, 0.4, 0.6, 0.8) ceramics as functions of sintering temperature

(ε_r = 17.6),[11] the $SrLaGaO_4$-$SrLa(Mg_{0.5}Ti_{0.5})O_4$ solid solution with near-zero τ_f is also of a higher dielectric constant than the $SrLaAlO_4$-based counterpart (22.2 for $0.35SrLaAlO_4$-$0.65SrLa(Mg_{0.5}Ti_{0.5})O_4$).[18]

3.2 | $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics

Figure 6 shows the relative density of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2, 0.3, 0.4, 0.6, 0.8) ceramics as a function of sintering temperature. With the increase in sintering temperature, the relative density first increases and then decreases for x = 0.2 and 0.3, and the highest density is obtained at 1350°C. However, the relative density increases monotonically with increase in the sintering temperature up to 1375°C for x = 0.4-0.8, and melting is observed on the surfaces of the products sintered at higher temperatures. Therefore, the highest sintering temperature of 1375°C is adopted for x = 0.4-0.8. The highest relative density over 97% is obtained for all the compositions.

Figure 7 shows the SEM images on the thermally etched surfaces of the $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics sintered at 1350°C. Similar to the $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics, the grain size also trends to increase with x. The inhomogenous microstructure with bright particles at the grain boundaries are observed for x = 0.3, 0.6, and 0.8. This is similar to $SrLa(Zn_{0.5}Ti_{0.5})O_4$ ceramics sintered at 1400°C,[16] and is attributed to the volatilization of ZnO.

The XRD patterns of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics sintered at 1350°C are shown in Figure 8. All the diffraction peaks match the characteristic peaks of

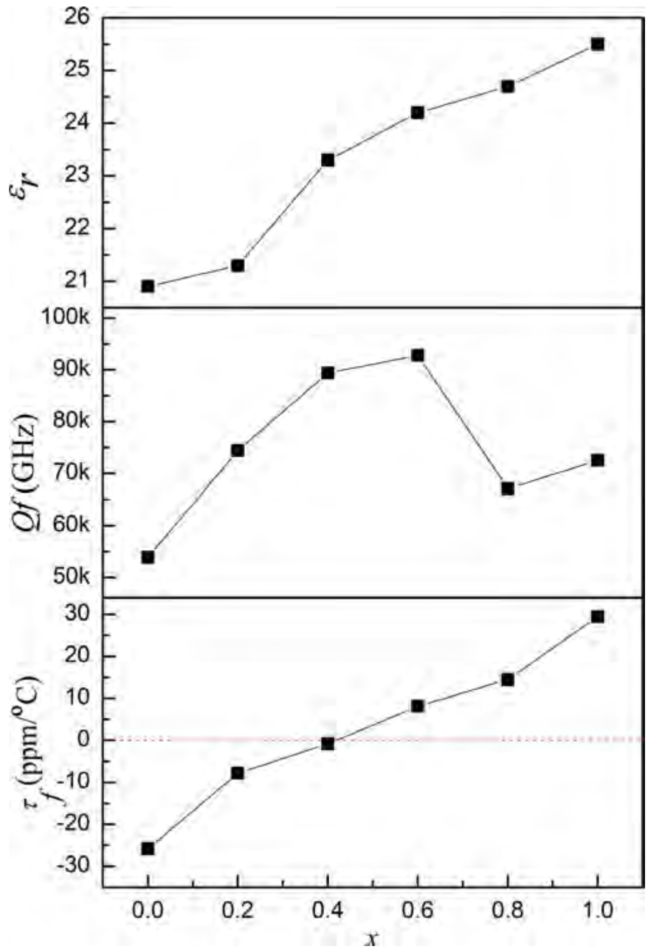

FIGURE 5 Optimal microwave dielectric properties of $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ (x = 0, 0.2, 0.4, 0.6, 0.8, 1.0) ceramics as functions of x

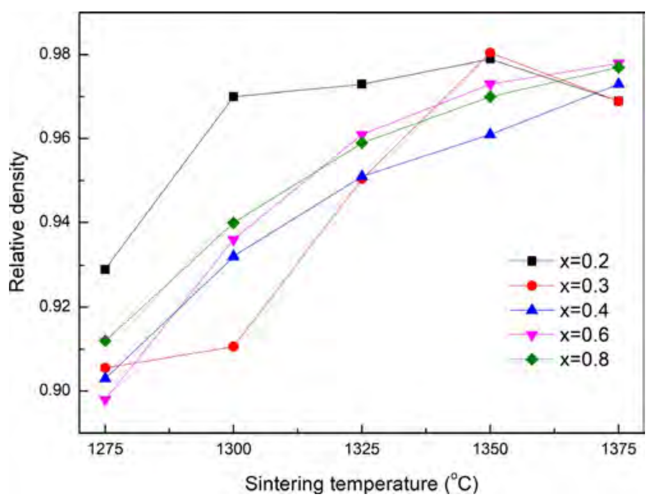

FIGURE 6 Relative density of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2, 0.3, 0.4, 0.6, 0.8) ceramics as a function of sintering temperature

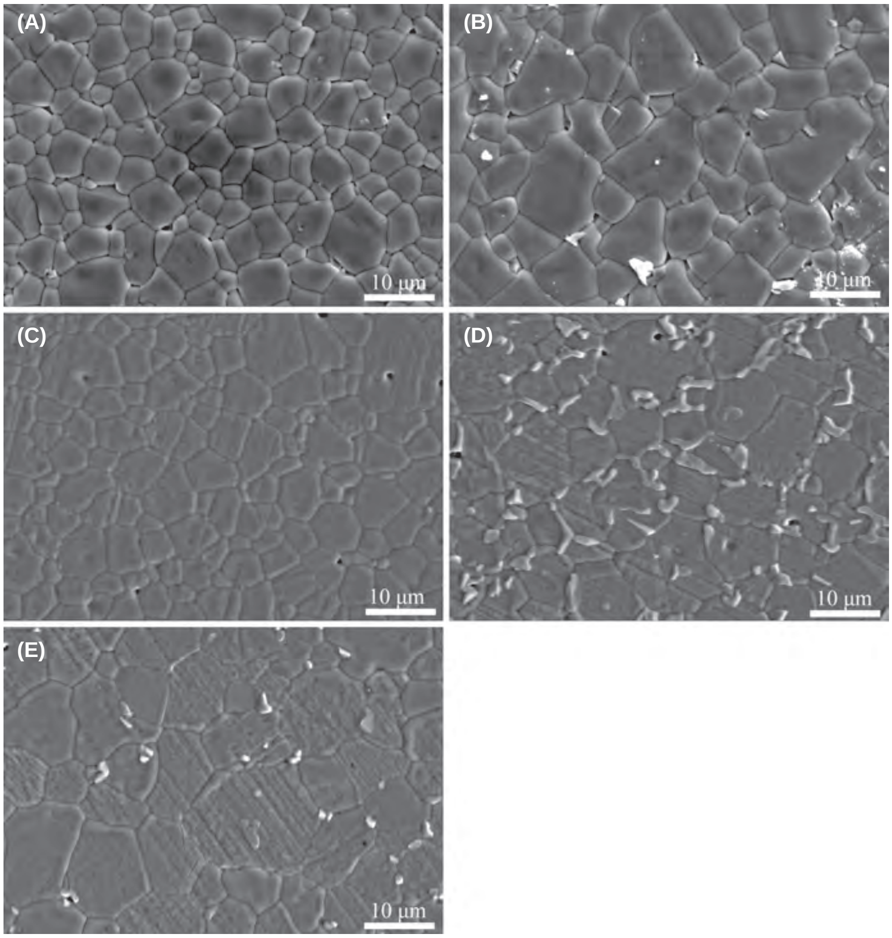

FIGURE 7 SEM micrographs of thermally etched surfaces for $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics: (A) $x = 0.2$, (B) $x = 0.3$, (C) $x = 0.4$, (D) $x = 0.6$ and (E) $x = 0.8$ sintered at 1350°C

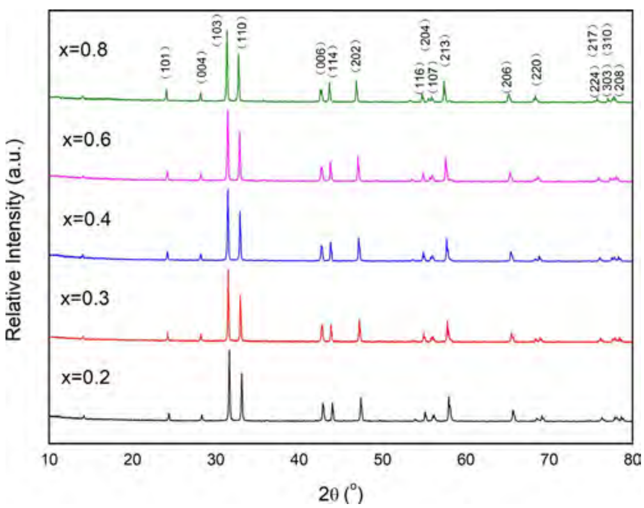

FIGURE 8 X-ray diffraction patterns of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ($x = 0.2$, 0.3, 0.4, 0.6, 0.8) ceramics

$SrLaGaO_4$ (JCPDS#80-1860), indicating the single phase with K_2NiF_4-type structure and *I*4/mmm space group for $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics. The XRD results further verify the prediction on the $SrLaGaO_4$-$SrLa(Zn_{0.5}Ti_{0.5})O_4$ unlimited solid solution by Ren et al.[16] The secondary phase is not revealed by XRD, indicating that the inhomogenous microstructure observed from SEM images should be formed on the surface during thermal etching, and the volatilization of ZnO during sintering inside the ceramic body is too slight to be detected by XRD.

Figure 9 shows the microwave dielectric properties of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics as functions of sintering temperature. The dielectric constant first rises and then fluctuates slightly for all the compositions, and this is consistent with the density shown in Figure 6. The *Qf* value decreases after peaking at a high value for $x = 0.2$-0.6, while it fluctuates mildly between 44 000 and 46 000 GHz for $x = 0.8$, which is similar to Mg-based counterpart. The sintering temperature for the highest *Qf* value is lower than that for the highest relative density for all compositions, and this is also partially attributed to the increasing concentration of oxygen vacancies with sintering temperature. Another possible reason is the slight volatilization of ZnO,[27,28] which is dependent on sintering temperature and has a more negative effect on the *Qf* value for higher sintering temperatures. Compared with ε_r and *Qf*, τ_f is insensitive to the sintering temperature.

Figure 10 shows the optimal microwave dielectric properties of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics as functions of *x*. With *x* increasing from 0 to1, the dielectric constant

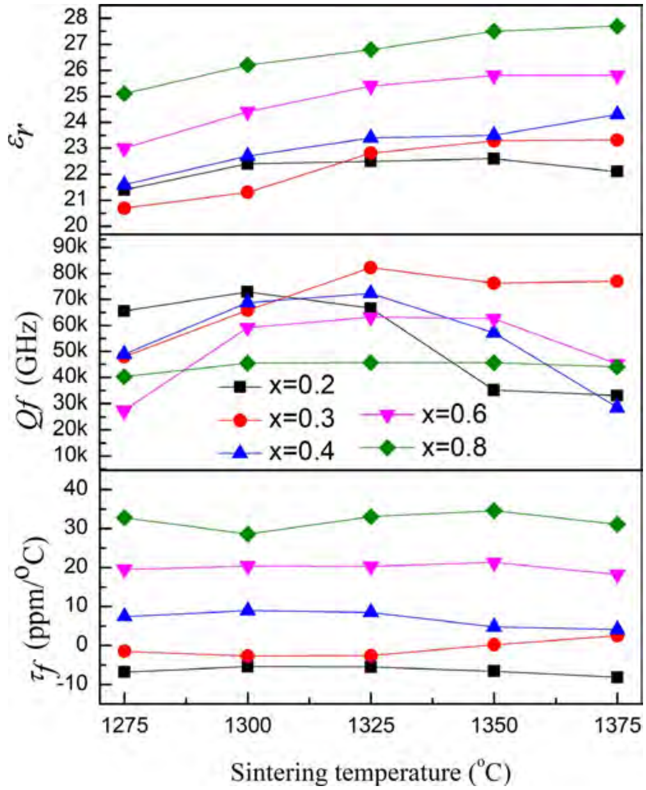

FIGURE 9 Microwave dielectric properties of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ (x = 0.2, 0.3, 0.4, 0.6, 0.8) ceramics as functions of sintering temperature

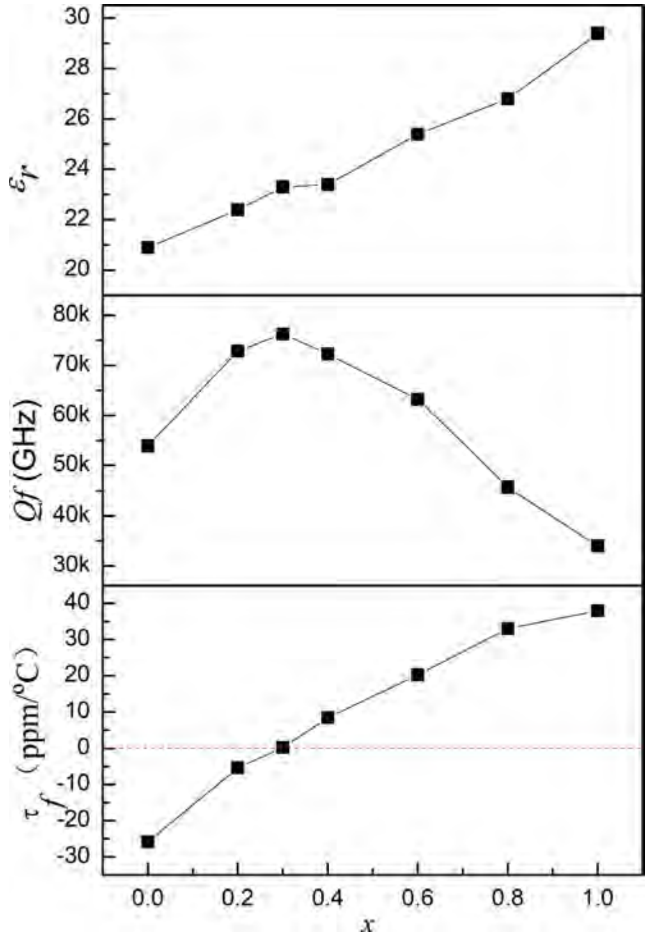

FIGURE 10 Optimal microwave dielectric properties of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ (x = 0, 0.2, 0.3, 0.4, 0.6, 0.8, 1.0) ceramics as functions of x

increases monotonically from 20.9 to 29.4, and the temperature coefficient of resonant frequency increases from −25.8 to 38 ppm/°C. Similar to the Mg-based ceramics, the Qf value of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics first increases from 53 900 to 76 300 GHz with increasing x from 0 to 0.3, and then decreases gradually to 34 000 GHz for x = 1. The good combination of microwave dielectric properties with ε_r = 23.3, Qf = 76 200 and τ_f = +0.2 ppm/°C are obtained for $SrLa[Ga_{0.7}(Zn_{0.5}Ti_{0.5})_{0.3}]O_4$ ceramics sintered at 1350°C.

Comparing with the $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ ceramics, the $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics are of significantly lower Qf values, especially for the Mg- and Zn-rich compositions. This is due to the significantly higher Qf value of the end-member $SrLa(Mg_{0.5}Ti_{0.5})O_4$ (72 000 GHz) than that of $SrLa(Zn_{0.5}Ti_{0.5})O_4$ (34 000 GHz).[16] It is well known that the Qf value of ceramics is determined by the intrinsic and extrinsic factors. Noting that the ion radius of Zn^{2+} (0.74 Å) is higher than that of Mg^{2+} (0.72 Å), the mismatch between the perovskite layer and rock salt layer is different for $SrLa[Ga_{1-x}(Mg_{0.5}Ti_{0.5})_x]O_4$ and $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ ceramics, which can be evaluated by the tolerance factor of the perovskite layer and may lead to the different Qf values. However, the tolerance factors are 0.917 and 0.914 for the end-members $SrLa(Mg_{0.5}Ti_{0.5})O_4$ and $SrLa(Zn_{0.5}Ti_{0.5})O_4$, respectively, and the slight difference is not enough for explaining the large difference between their Qf values. Another possible reason for the lower Qf value of $SrLa[Ga_{1-x}(Zn_{0.5}Ti_{0.5})_x]O_4$ is the volatilization of ZnO during sintering, which is usually slight but has a significant negative effect on the Qf value.[27,28]

4 | CONCLUSION

The dense $SrLa[Ga_{1-x}(R_{0.5}Ti_{0.5})_x]O_4$ (R = Mg, Zn) ceramics were prepared by a standard solid state sintering method. The single-phase ceramics with K_2NiF_4-type layered perovskite structure and $I4/mmm$ space group were obtained, indicating that $SrLaGaO_4$ and $SrLa(R_{0.5}Ti_{0.5})O_4$ can form the unlimited solid solutions as predicted. The sintering temperature for the highest Qf value is usually lower than that for the highest relative density, which is attributed to the increasing concentration of oxygen vacancies and volatilization of ZnO (only for Zn-based ceramics) with the sintering temperature. ε_r and τ_f increase monotonously with increasing x, while the Qf value first increases and then decreases. Due to the opposite τ_f values of $SrLaGaO_4$ and $SrLa(R_{0.5}Ti_{0.5})O_4$, the τ_f is successfully counteracted in the solid solutions. The optimized microwave dielectric properties were obtained as following: ε_r = 23.3, Qf = 89 400 GHz, τ_f = −0.8 ppm/°C for $SrLa[Ga_{0.6}(Mg_{0.5}Ti_{0.5})_{0.4}]O_4$ and ε_r = 23.3, Qf = 76 200 GHz, τ_f = 0.2 ppm/°C for $SrLa[Ga_{0.7}(Zn_{0.5}Ti_{0.5})_{0.3}]O_4$, indicating that the present solid solution ceramics are the promising candidates as microwave resonator materials for the telecommunication applications.

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