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Microwave dielectric properties of $BaO–2(1-x)ZnO–xNd_2O_3–4TiO_2$ ($x = 0–1.0$) ceramics

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Abstract

Microwave dielectric properties of $(1-x)BaZn_2Ti_4O_{11}–xBaNd_2Ti_4O_{12}$ ($x = 0–1.0$) ceramics were investigated by the solid-state reaction with the purpose of finding a microwave ceramics with high dielectric constant (ε_r), high quality factor ($Q \times f$) and low temperature coefficient of resonant frequency (τ_f). A two phase system $BaZn_2Ti_4O_{11}–BaNd_2Ti_4O_{12}$ was formed and SEM photographs show equiaxed $BaZn_2Ti_4O_{11}$ grains and columnar $BaNd_2Ti_4O_{12}$ grains. The microwave dielectric properties were strongly determined by the chemical composition. As increasing x from 0 to 1.0, the phase composition varied from pure $BaZn_2Ti_4O_{11}$, to the two phase system $BaZn_2Ti_4O_{11}–BaNd_2Ti_4O_{12}$ and then to pure $BaNd_2Ti_4O_{12}$. Therefore, the ε_r raised from 29.1 to 82.0 and the $Q \times f$ values decreased from 54,630 GHz to 8110 GHz, and the τ_f values increased from −29 ppm/°C to 94 ppm/°C. $0.8BaZn_2Ti_4O_{11}–0.2BaNd_2Ti_4O_{12}$ ceramics sintered at 1250 °C for 2.5 h had $\varepsilon_r = 39.1$, $Q \times f = 37{,}850$ GHz and $\tau_f = -9$ ppm/ °C.

Keywords: A. Sintering; C. Dielectric properties; Microwave ceramics

1. Introduction

With the operating frequency of mobile telecommunications expanding to microwave frequency, creation of microwave ceramics with a high dielectric constant (ε_r), high quality factor ($Q \times f$) and a near zero temperature coefficient of resonant frequency (τ_f) in wide temperature and frequency ranges has been a very active research field in the past few decades [1–3]. The application of such functional ceramics ensures the miniaturization and the cost reduction of microwave equipment. In particular, the miniaturization is mainly a result of the use of high dielectric constant materials because of the physical length of a dielectric resonator $\approx 1/\sqrt{\varepsilon_r}$. High quality factor is good for microwave selectivity and a near zero temperature coefficient of resonant frequency is one of the major requirements for dielectric materials to be utilized in a frequency-stable passive component [4].

Middle dielectric constant ceramics based on BaO–ZnO–TiO_2 (BZT) system with lower sintering temperature than $BaTi_4O_9/Ba_2Ti_9O_{20}$ and adjustable temperature coefficient of resonate frequency has been researched. Roth et al. had reported BZT system has four ternary phases: $Ba_4ZnTi_{11}O_{27}$, $BaZn_2Ti_4O_{11}$, $Ba_2ZnTi_5O_{13}$ and hollandite-type solid solutions ($Ba_xZn_xTi_{8-x}O_{16}$) [5]. And Zhou et al. [6] reported the microwave dielectric properties of $Ba_4ZnTi_{11}O_{27}$ ceramics ($\varepsilon_r \approx 36.8$, $Q \times f = 16{,}460$ GHz and $\tau_f = 17.2$ ppm/°C). Belous et al. researched the homogeneity range and the microwave dielectric properties of $BaZn_2Ti_4O_{11}$ ceramics ($\varepsilon_r \approx 30$, $Q \times f = 68{,}000$ GHz and $\tau_f = -30$ ppm/°C) [7]. One attraction is that the $Q \times f$ value of $BaZn_2Ti_4O_{11}$ is much higher than that of $BaTi_4O_9$ ($\varepsilon_r = 37$, $Q \times f = 22{,}700$ GHz and $\tau_f = 15$ ppm/°C) and $Ba_2Ti_9O_{20}$ ($\varepsilon_r = 39$, $Q \times f = 32{,}000$ GHz and $\tau_f = 2$ ppm/°C) [8,9]. On the other hand, $BaO–Nd_2O_3–TiO_2$ ceramics with high dielectric constant has also been widely studied for microwave applications. Ceramics, with $BaO:Nd_2O_3:TiO_2$ of around 1:1:4 and a high dielectric constant ($\varepsilon_r = 70–90$), has been commonly applied in mobile telephone systems [10]. The chemical compound $BaNd_2Ti_4O_{12}$, in particular, had emerged as a good microwave ceramics because it exhibits high dielectric constant ($\varepsilon_r = 84$) and high quality factor ($Q \times f = 7800$ GHz) [11]. But its large positive temperature coefficient of resonant frequency ($\tau_f \geq 140$ ppm/°C) [10] makes it imperfect for the use as dielectric resonator.

Usually, there are two methods to obtain a microwave dielectric ceramics with a near-zero temperature coefficient.

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One is to form a solid solution [12], and the other one is to combine two chemical compounds with opposite temperature coefficients to form the composite ceramics [13,14]. According to the second idea and taking the microwave dielectric properties of $BaZn_2Ti_4O_{11}$ and $BaNd_2Ti_4O_{12}$ as reference, we designed this chemical composition $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ with $x = 0$–1.0 for research. $BaZn_2Ti_4O_{11}$ and $BaNd_2Ti_4O_{12}$ might co-exist because these two phases have two same main chemical elements Ba and Ti. In addition, ZnO might lower the sintering temperature and increase the $Q \times f$ value of BaO–Nd_2O_3–TiO_2 ceramics, and Nd_2O_3 might increase the dielectric constant of BaO–ZnO–TiO_2 ceramics. In this paper, the effect of sintering temperature and chemical composition on the structural stability and microwave dielectric properties of BaO–ZnO–Nd_2O_3–TiO_2 ceramics was investigated and a new microwave ceramics with a near zero temperature coefficient was obtained at last.

2. Experimental

2.1. Sample preparation

The samples used in this study were prepared by the conventional solid-state reaction technique. For the synthesis of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ($x = 0$–1.0) powders, $BaCO_3$ (Xilong Group, Shantou), ZnO (Xilong Group, Shantou), Nd_2O_3 (Jichang Rare Earth Co. Ltd., Zhuhai) and TiO_2 (Xilong Group, Shantou) of >99% purity were mixed and ball-milled in a nylon jar with zirconia balls and deionized water for 20 h, and after drying these powders were calcined at 750–1000 °C for 3 h. Then, with 7 wt% acrylicacid solution as binder, the calcined powders were pressed into cylindrical samples with 18 mm in diameter and 7 mm in thickness under a pressure of 250 kg/cm^2. At last, these samples were sintered at 1150–1325 °C for 2.5 h.

2.2. Characterization

The bulk densities of the sintered samples were measured by the Archimedes method. The phase composition was identified by X-ray diffraction (XRD) using CuKα radiation (DX–1000 CSC). Microstructure observation was conducted on the surface of samples by using scanning electron microscopy (SEM, FEI Inspect F). The dielectric characteristics at microwave frequencies were measured by the Hakki–Coleman dielectric resonator method in the TE_{011} mode using a network analyzer (HP83752A) [15]. The sintered samples used for this measurement had the ratio: diameter/height = 1.9–2.3. The τ_f value was determined from the difference between the resonant frequencies obtained at 25 °C and 80 °C using the equation: $\tau_f = (f_{t_2} - f_{t_1})/(f_{t_1} \times (t_2 - t_1))$, where f_{t_1} and f_{t_2} are the resonant frequencies at $t_1 = 25$ °C and $t_2 = 80$ °C, respectively.

3. Results and discussion

Fig. 1(a)–(c) shows the X-ray diffraction patterns of $0.6BaZn_2Ti_4O_{11}$–$0.4BaNd_2Ti_4O_{12}$ powders calcined at

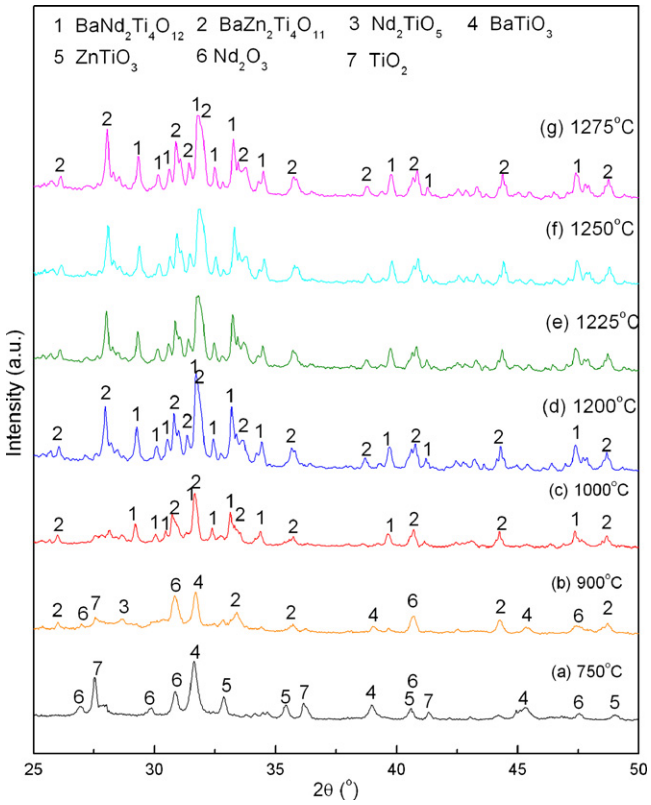

Fig. 1. XRD patterns of $0.6BaZn_2Ti_4O_{11}$–$0.4BaNd_2Ti_4O_{12}$ powders calcined at (a) 750 °C, (b) 900 °C and (c) 1000 °C for 3 h and ceramics sintered at different temperatures for 2.5 h.

750–1000 °C for 3 h. When the calcining temperature was at 750 °C, two raw materials Nd_2O_3 and TiO_2 and two newly formed phases $BaTiO_3$ and $ZnTiO_3$ were observed. As the calcining temperature was increased to 900 °C, $BaZn_2Ti_4O_{11}$ began to form by the reaction along TiO_2, $BaTiO_3$ and $ZnTiO_3$, and for $BaNd_2Ti_4O_{12}$, it was not formed at this temperature but a precursor phase Nd_2TiO_5 had been formed. When continuing increasing the temperature to 1000 °C, $BaNd_2Ti_4O_{12}$ was formed and thus a two phase system $BaZn_2Ti_4O_{11}$–$BaNd_2Ti_4O_{12}$ was formed. The $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics was produced at sintering temperatures 1150–1325 °C for 2.5 h. Fig. 1(d)–(g) presents the X-ray diffraction patterns of $0.6BaZn_2Ti_4O_{11}$–$0.4BaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures. The XRD patterns show the two phase system $BaZn_2Ti_4O_{11}$–$BaNd_2Ti_4O_{12}$ which was remarkably stable at different sintering temperatures. This property is extremely useful if these composite ceramics are applied in the real production. Meanwhile, the effect of the compositional ratio on the phase composition was also investigated. Fig. 2 shows the XRD patterns of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at their own optimal temperatures. At $x = 0$ and $x = 1.0$, pure $BaZn_2Ti_4O_{11}$ phase and $BaNd_2Ti_4O_{12}$ phase were observed, respectively. When x was increased from 0.1 to 0.9, $BaZn_2Ti_4O_{11}$ and $BaNd_2Ti_4O_{12}$ co-existed with each other and formed a two phase system, and $BaZn_2Ti_4O_{11}$ gradually decreased and $BaNd_2Ti_4O_{12}$ gradually accumulated. It was easy to understand that the different ionic radius and valence between Zn^{2+} and Nd^{3+}

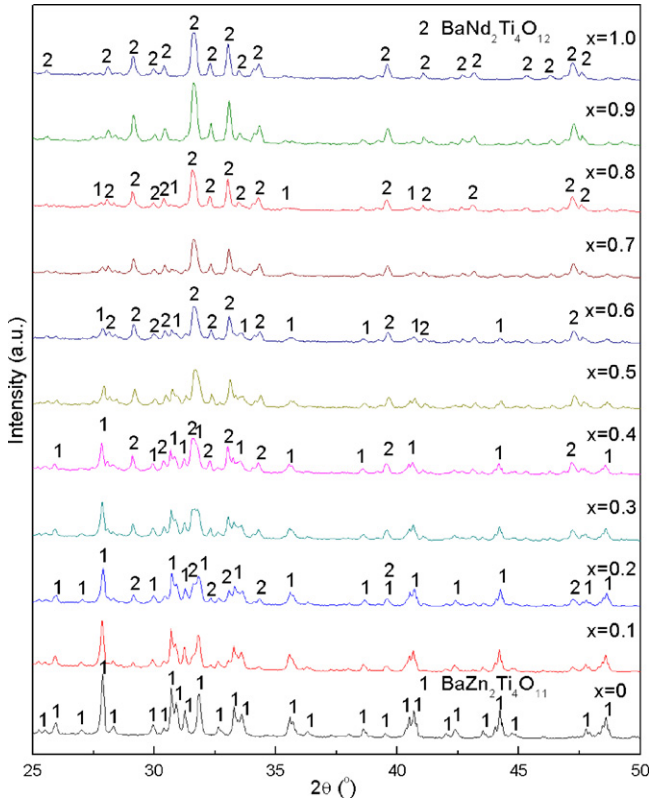

Fig. 2. XRD patterns of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at their own optimal temperatures for 2.5 h.

made the formation of solid solution difficult and there was also no reaction between $BaZn_2Ti_4O_{11}$ and $BaNd_2Ti_4O_{12}$. Thus, it was reliable to control the phase composition by the compositional ratio.

To investigate the relationship between the sintering temperature and the microstructure, the SEM photographs for $0.6BaZn_2Ti_4O_{11}$–$0.4BaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures for 2.5 h are shown in Fig. 3. When increasing the sintering temperature from 1200 to 1275 °C, two kinds of crystal growth were exhibited. One was that small grains grew uniformly into equiaxed grains and the other one was that small grains grew along a preferred orientation forming columnar grains. At the same time, pores being obvious at 1200 °C (Fig. 3(a)) were gradually eliminated until 1250 °C (Fig. 3(c)) and the ceramics sintered at 1250 °C and 1275 °C shown compact microstructure with crystal grains in dense contact. Especially when at 1275 °C, a few columnar grains were inset into equiaxed grains because of the softening of equiaxed grains at this high sintering temperature, for example, the marked areas in Fig. 3(d).

Fig. 4 displays the effect of the compositional ratio on the microstructure. For identifying the two kinds of grains, energy-dispersive X-ray (EDX) analysis was used in Fig. 4(b) and the results are described in Table 1. From the EDX results the large equiaxed grains were identified as $BaZn_2Ti_4O_{11}$ and the small columnar grains were $BaNd_2Ti_4O_{12}$. As could be seen from the micrographs, it was clear at a glance that as x was increased, the

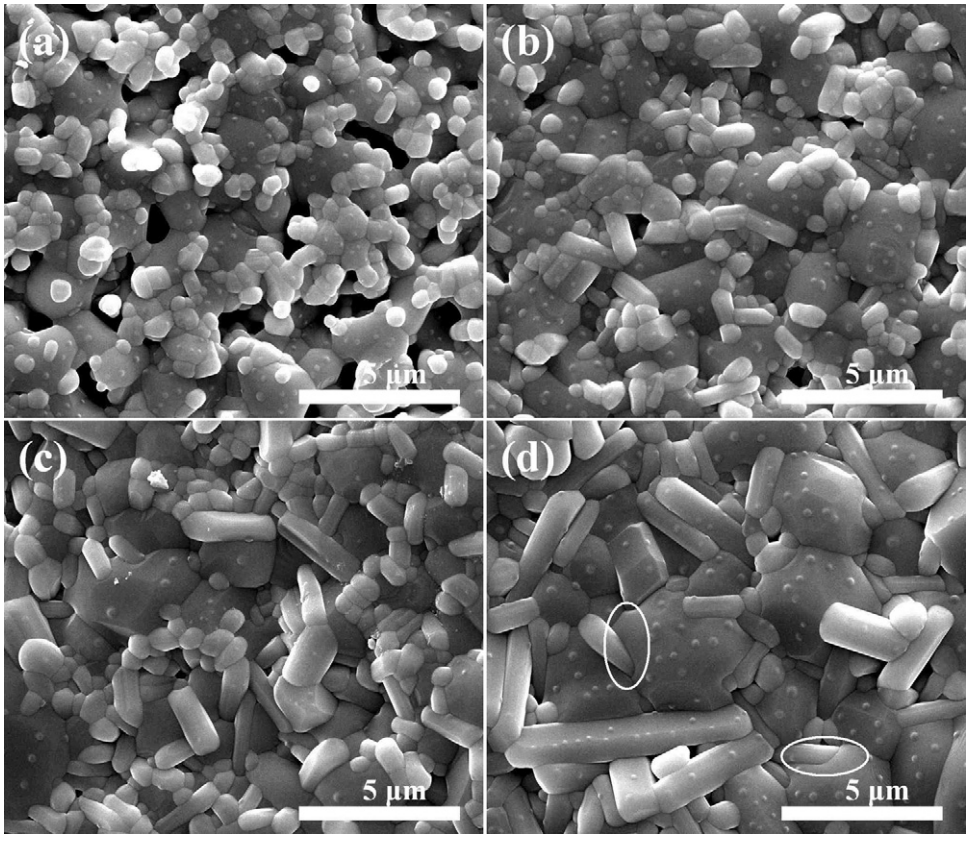

Fig. 3. SEM photographs of $0.6BaZn_2Ti_4O_{11}$–$0.4BaNd_2Ti_4O_{12}$ ceramics sintered at (a) 1200 °C, (b) 1225 °C, (c) 1250 °C and (d) 1275 °C for 2.5 h.

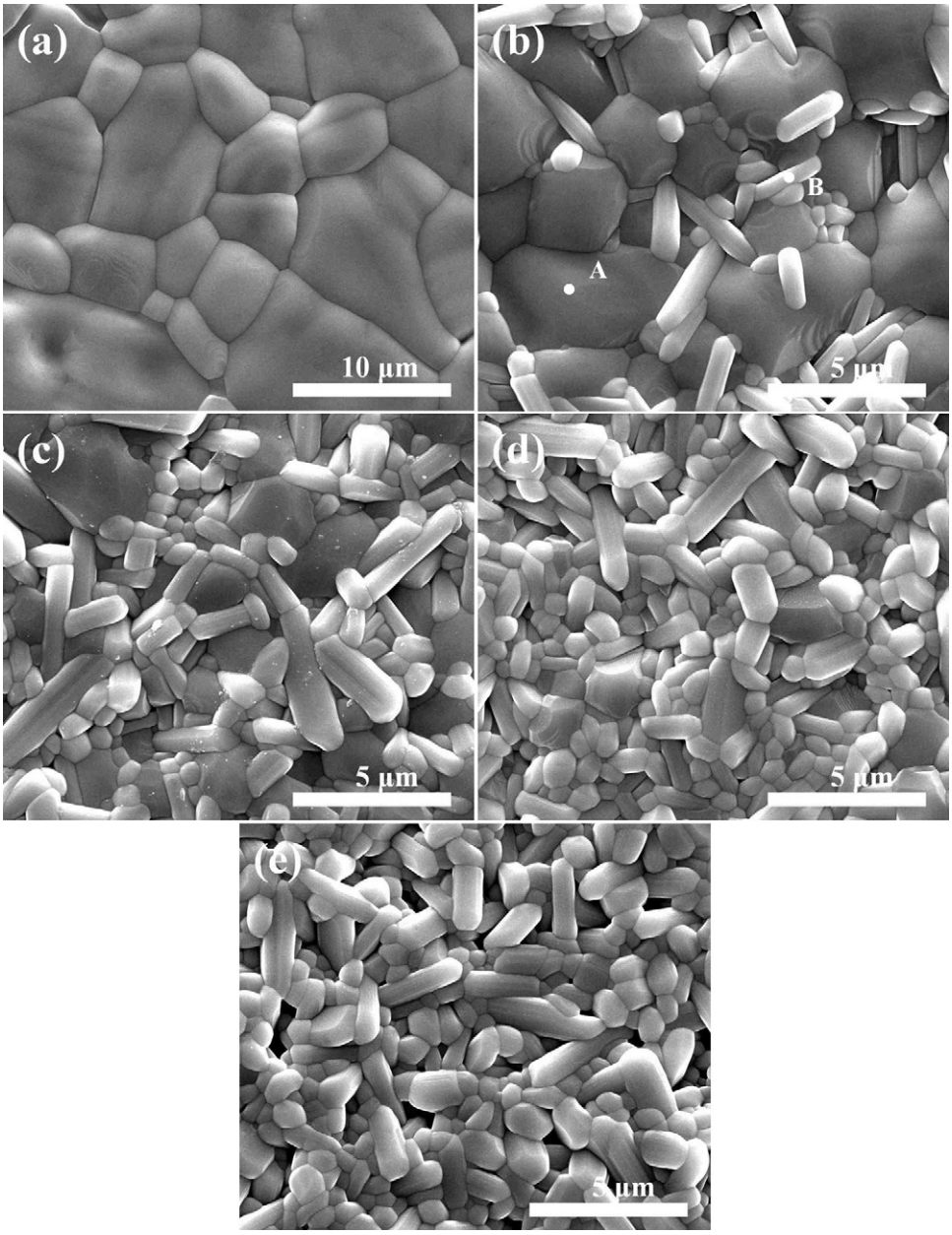

Fig. 4. SEM photographs of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics with (a) $x = 0$, 1200 °C, (b) $x = 0.2$, 1250 °C, (c) $x = 0.5$, 1250 °C, (d) = 0.8, 1275 °C and (e) $x = 1.0$, 1300 °C for 2.5 h.

volume percentage of $BaZn_2Ti_4O_{11}$ grains declined continuously and by contrast $BaNd_2Ti_4O_{12}$ grains grew gradually. At $x = 0$, pure $BaZn_2Ti_4O_{11}$ ceramics sintered at 1200 °C had a dense microstructure and the abnormal grain growth caused the maximum grain size above 10 μm. While x was changing from 0.1 to 0.9, the abnormal grain growth of $BaZn_2Ti_4O_{11}$ was restrained due to the pinning effect of $BaNd_2Ti_4O_{12}$ grains in the grain boundary, and thus the maximum grain size of $BaZn_2Ti_4O_{11}$ was limited to less than 5 μm. When $x = 1.0$, pure $BaNd_2Ti_4O_{12}$ ceramics sintered at 1300 °C still had certain pores because its sintering temperature was usually above 1350 °C [16]. The above results implied that the densification of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics at 1200–1300 °C was mainly attributed to the lower sintering temperature (about 1200 °C) of $BaZn_2Ti_4O_{11}$ phase.

Table 1
The energy dispersive X-ray analysis (EDX) data of $0.8BaZn_2Ti_4O_{11}$–$0.2BaNd_2Ti_4O_{12}$ ceramics corresponding to Fig. 4(b).

Spot	Atom (%)				
	Ba	Zn	Nd	Ti	O
A	7.35	14.13	0.82	28.26	49.44
B	8.24	1.11	15.27	31.44	43.94

Fig. 5 shows the bulk density of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures for 2.5 h. For all these samples except the sample with $x = 1.0$, when increasing the sintering temperature their densities

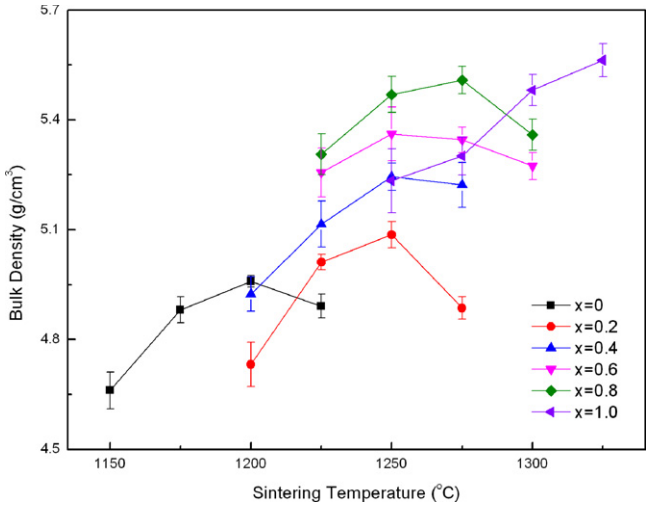

Fig. 5. The bulk density of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures for 2.5 h.

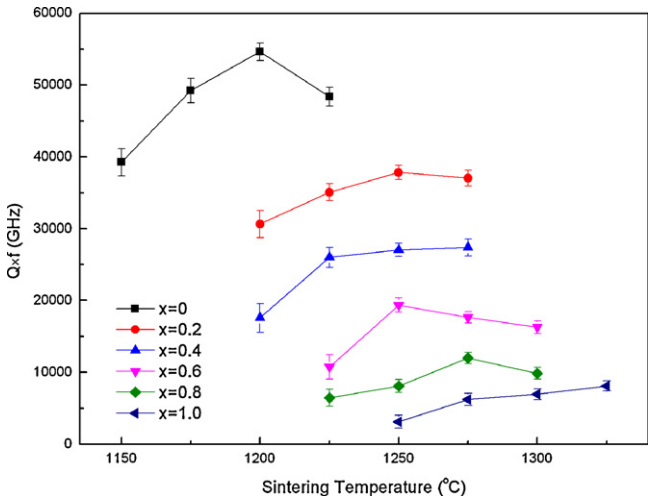

Fig. 7. The $Q \times f$ value of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures for 2.5 h.

increased noticeably at first, which was attributed to the elimination of pores, and then started to decline after reaching their maximum mainly because of the abnormal grain growth as shown in Fig. 3. For the pure $BaNd_2Ti_4O_{12}$ sample, its density kept increasing till 1325 °C, which was similar with the report in the literature [11]. Moreover, the sintering temperature could be effectively lowered with increasing the ZnO content because $BaZn_2Ti_4O_{11}$ (about 1200 °C) had lower sintering temperature than $BaNd_2Ti_4O_{12}$ (above 1350 °C). And the maximum densities of different samples approximately increased linearly with increasing x value because $BaNd_2Ti_4O_{12}$ phase with a higher density occupied more and more volume in the two phase system.

Fig. 6 shows the dielectric constant (ε_r) of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics as a function of the sintering temperature. The variation tendency of ε_r was closely in accord with the trend between the bulk density and the sintering temperature because both of the density and dielectric permittivity are influenced much by the pores (density ≈ 0 and $\varepsilon_r \approx 1$) in samples before the ceramics is well sintered. For example, the ε_r value for the sample with $x = 0.2$ increased from 35.1 at 1200 °C to 39.1 at 1250 °C and then declined to 38.4 at 1275 °C. What is more, the ε_r value was strongly dependent on the x value. When x was changed from 0 to 1.0, the ε_r value increased sharply from 29.1 to 81.9. This was primarily because $BaNd_2Ti_4O_{12}$ phase possessing a higher dielectric constant increased with increasing x value. The ε_r values on both ends of the coordinate axis for pure $BaZn_2Ti_4O_{11}$ and $BaNd_2Ti_4O_{12}$ samples were found to be compatible with earlier reports [7,10], and the ε_r values in the middle, therefore, should be credible.

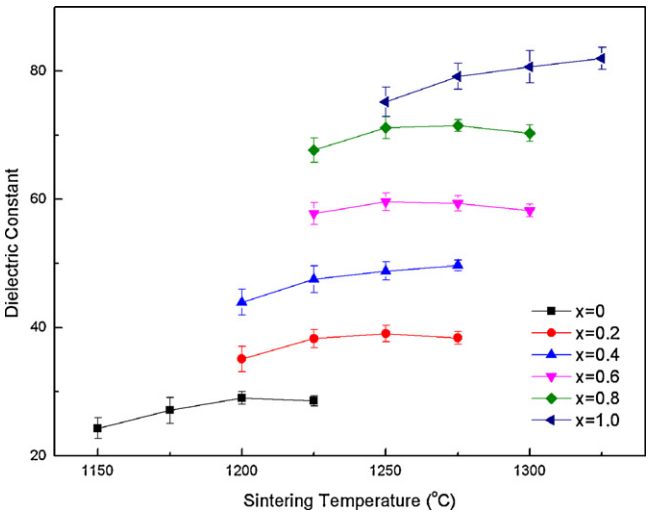

Fig. 6. The dielectric constant of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures for 2.5 h.

The quality factor value ($Q \times f$) of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures for 2.5 h is shown in Fig. 7. The $Q \times f$ value is usually affected by many factors such as lattice vibrational modes, pores, second phases, impurities, lattice defect, crystallizability, cation ordering and inner stress, so sometimes it is difficult to determine the key influencing factor [17]. When increasing the sintering temperature, the $Q \times f$ values of the samples with $x = 0$–0.8 were increased initially, then declined or saturated ($x = 0.4$) thereafter, but for $x = 1.0$, the $Q \times f$ value strongly raised till 1325 °C. It was reasonably believed that these changes were related to the samples' compactness and usually, the denser the microstructure is, the higher the $Q \times f$ value is. On the other hand, when increasing x from 0 to 1.0, the $Q \times f$ values maintained a downward trend but dropped slower and slower, for instance, from 54,630 GHz ($x = 0$) to 37,850 GHz ($x = 0.2$) and from 11,980 GHz ($x = 0.8$) to 8110 GHz ($x = 1.0$). Since the $Q \times f$ value of $BaNd_2Ti_4O_{12}$ was much lower than that of $BaZn_2Ti_4O_{11}$, the $Q \times f$ values of different samples declined with increasing x value. That the $Q \times f = 54{,}630$ GHz for pure $BaZn_2Ti_4O_{11}$ was smaller than 68,000 GHz reported by Belous et al. [7] should be a result of inhomogeneous microtopography and the $Q \times f = 8110$ GHz for pure $BaNd_2Ti_4O_{12}$ accorded with the reported results [10,16].

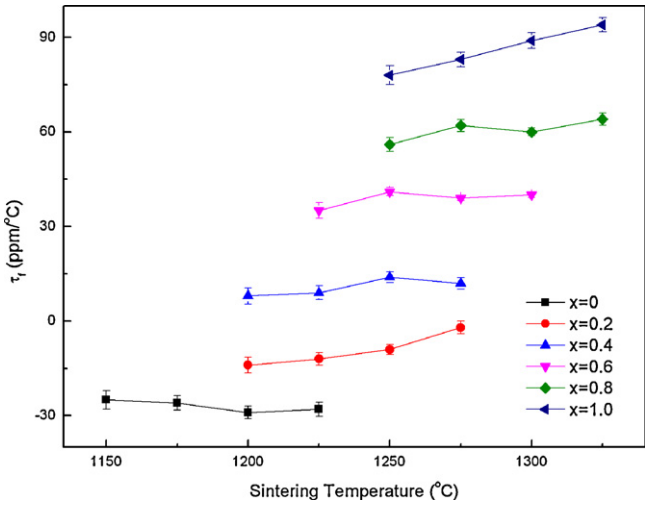

Fig. 8. The temperature coefficient of resonant frequency of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics sintered at different temperatures for 2.5 h.

The temperature coefficient of resonant frequency (τ_f) of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics as a function of the sintering temperature is illustrated in Fig. 8. The τ_f value of a composite ceramics basically depends on the phase composition. As shown in Fig. 8, when x was increased from 0 to 1.0, the τ_f value varied linearly in a positive direction from −29 ppm/°C at $x = 0$ to 94 ppm/°C at $x = 1.0$. When $x = 0.2$ and 0.4, the τ_f values for the well sintered samples were −9 ppm/°C and 14 ppm/°C respectively, which suggested that the microwave ceramics with a zero τ_f value in this system could be synthesized with x between 0.2 and 0.4. In principle, the reason for the tuning of τ_f to a near zero value should be the mutual offset effect between $BaZn_2Ti_4O_{11}$ ($\tau_f = -29$ ppm/°C) and $BaNd_2Ti_4O_{12}$ ($\tau_f = 94$ ppm/°C or above 140 ppm/°C by Fu et al. [10]). Moreover the sintering temperature had little effect on the τ_f value due to no significant change of phase composition was observed at different sintering temperatures.

4. Conclusions

In order to find a microwave dielectric ceramics with high ε_r, $Q \times f$ and a near-zero τ_f, the sintering behavior and microwave dielectric properties of $(1-x)BaZn_2Ti_4O_{11}$–$xBaNd_2Ti_4O_{12}$ ceramics were investigated. A two phase system $BaZn_2Ti_4O_{11}$–$BaNd_2Ti_4O_{12}$ was formed and it was stable at different sintering temperatures. SEM photographs show equiaxed $BaZn_2Ti_4O_{11}$ grains and columnar $BaNd_2Ti_4O_{12}$ grains. The microwave dielectric properties were strongly determined by the chemical composition. As x was increased from 0 to 1.0, the phase composition varied from pure $BaZn_2Ti_4O_{11}$ phase, to the two phase system $BaZn_2Ti_4O_{11}$–$BaNd_2Ti_4O_{12}$ and then to pure $BaNd_2Ti_4O_{12}$ phase. Therefore, the ε_r raised from 29.1 to 82.0 and the $Q \times f$ value decreased from 54,630 GHz to 8110 GHz, and the τ_f value increased from −29 ppm/°C to 94 ppm/°C. $0.8BaZn_2Ti_4O_{11}$–$0.2BaNd_2Ti_4O_{12}$ ceramics sintered at 1250 °C for 2.5 h had $\varepsilon_r = 39.1$, $Q \times f = 37{,}850$ GHz and $\tau_f = -9$ ppm/°C, which were comparable with the dielectric properties of $BaTi_4O_9$ and $Ba_2Ti_9O_{20}$.

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