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Effect of Bi_2O_3 and B_2O_3 additives on the sintering temperature, microstructure, and microwave dielectric properties for $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics

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Abstract

The microwave dielectric properties of $Sm(Mg_{0.5}Ti_{0.5})O_3$ incorporated with various amount of Bi_2O_3 and B_2O_3 additives have been investigated systematically. In this study, both Bi_2O_3 and B_2O_3 additives acting as a sintering aid can effectively lower the sintering temperature from 1550 °C to 1300 °C. The ionic radius of Bi^{3+} for a coordination number of 6 is 0.103 nm, whereas the ionic radius of B^{3+} is 0.027 nm. Clearly, the ionic radius of Bi^{3+} is greatly larger than one of B^{3+}, which resulted in the specimens incorporated with Bi_2O_3 having larger lattice parameters and cell volume than those incorporated with B_2O_3. The experimental results show that no second phase was observed throughout the entire experiments. Depending on the interfacial tension, the liquid phase may penetrate the grain boundaries completely, in which case the grains will be separated from one another by a thin layer as shown in $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with Bi_2O_3. Whereas, in $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with B_2O_3, the volume fraction of liquid is high, the grains may dissolve into the liquid phase, and rapidly rearrange, in which case contact points between agglomerates will be dissolved due to their higher solubility in the liquid, leading plate-like shape microstructure.

A dielectric constant (ε_r) of 29.3, a high $Q \times f$ value of 26,335 GHz (at 8.84 GHz), and a τ_f of -32.5 ppm/°C can be obtained for $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with 10 mol% Bi_2O_3 sintered at 1300 °C. While $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with 5 mol% B_2O_3 can effectively lower temperature coefficient of resonant frequency, which value is -21.6 ppm/°C. The $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramic incorporated with heavily Bi_2O_3 and B_2O_3 additives exhibits a substantial reduction in temperature (~250 °C) and compatible dielectric properties in comparison with that of an un-doped one. This implied that this ceramic is suitable for miniaturization in the application of dielectric resonators and filters by being appropriately incorporated with a sintering aid.

Keywords: A. Powder: solid state reaction; A. Sintering; C. Dielectric properties

1. Introduction

Because of the rapid development in the microwave communication system, satellite broadcasting system as well as wireless mobile phone systems, it has become very important for the miniaturization of microwave devices such as oscillators, band pass filters, duplexers and global positioning systems (GPS) patch antennas [1,2]. To miniaturize the devices and for the systems to work with high efficiency and stability, the materials for microwave resonators must be excellent in the following three dielectric characteristics. The first characteristic is a high dielectric constant. The use of high dielectric constant materials can effectively reduce the size of resonators since the wavelength (λ) in dielectrics is inversely proportional to $\sqrt{\varepsilon_r}$ of the wavelength in vacuum (λ_0) ($\lambda = \lambda_0/\sqrt{\varepsilon_r}$). The second is a high quality factor ($Q \times f$) value. This is required to achieve high frequency selectivity and stability in microwave transmitter and receiver components. There are a number of factors affecting the microwave dielectric loss which can be divided into two kinds, the intrinsic loss and extrinsic loss. The intrinsic losses are mainly caused by crystal structure, while the extrinsic losses are dominated by second phases, oxygen

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vacancies, grain sizes and densification or porosity [3]. The third is a near zero temperature coefficient of resonant frequency (τ_f) for dielectric resonators and microwave device substrates [4,5]. Small temperature coefficients of the resonant frequency ensure the stability of the microwave components at different working temperatures. Using two or more compounds with negative and positive temperature coefficients to form a solid solutions or mixed crystal is the most promising method of obtaining a zero temperature coefficient of the resonant frequency. Because most dielectric ceramics with high dielectric constant have a positive τ_f value, searching for materials with a high dielectric constant, a high Q and a negative τ_f is necessary to achieve this goal [6].

Recently, $Ln(B_{0.5}Ti_{0.5})O_3$ (Ln = La, Sm, Nd; B = Mg, Zn) has been reported as low dielectric loss ($\tan\delta = 1/Q$) material and have a reasonable dielectric constant with an adjustable temperature coefficient of resonant frequency [7–10]. Among these perovskite compounds $Sm(Mg_{0.5}Ti_{0.5})O_3$ exhibited a high dielectric constant ($\varepsilon_r = 25$), a high quality factor ($Q \times f$ = 35,000 GHz) and a negative temperature coefficient at resonant frequency ($\tau_f = -26$ ppm/°C) [11]. For ideal density 6.6 g/cm^3, it needs very high sintering processing temperature to 1550 °C. Low temperature solid-state synthesis is an approach that shows great promise for the synthesis of materials with low cost. Usually, three methods are commonly used for reducing the sintering temperature of dielectric ceramics: low melting-temperature glass addition, chemical processing, and powder with smaller particle sizes [12–14]. The first method using liquid phase glass sintering was found to effectively lower the firing temperature. However, it also decreased the microwave dielectric properties of dielectric resonators, especially the quality factor. The chemical process often required a complex procedure, which was expensive and time consuming. Therefore, the selection of non-glass addition with a low melting point is extremely important.

Since Bi_2O_3 and B_2O_3 are commonly used as a liquid-phase flux and have been shown to accomplish a substantial sintering temperature reduction [15,16], they were selected as a sintering aid in the present study. The objective of this study is to develop $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics with low sintering temperature, high dielectric constant, a high quality factor and near to zero τ_f by incorporating different amount of Bi_2O_3 and B_2O_3 sintering aids. The resultant microwave dielectric properties were considered based upon the densification, the X-ray diffraction patterns and the microstructures of the ceramics.

2. Experimental procedure

Specimen powders were prepared by a conventional solid-state method. High-purity oxide powders (>99.9%): Sm_2O_3, MgO and TiO_2 were used as raw materials. The powders were weighed according to the composition $Sm(Mg_{0.5}Ti_{0.5})O_3$, and were ground in distilled water for 12 h in a balling mill with agate balls. Prepared powders were dried and calcined at 1100 °C for 2 h in air. The calcined powders were mixed as desired composition $Sm(Mg_{0.5}Ti_{0.5})O_3$ with different amounts of Bi_2O_3 and B_2O_3 additives as sintering aids and re-milled for 12 h. These fine powders were pressed into pellets with dimensions of 11 mm in diameter and 5.5 mm in thickness by pressing with 150 MPa. These pellets were then sintered at a temperature of 1300 °C for 6 h in air. The heating and cooling rates were both set at 5 °C/min.

The fracture surfaces of the sintered ceramics were observed by means of scanning electron microscopy (SEM, JEOL JSM 6400, Japan) and an energy-dispersive X-ray spectrometer. The crystalline phase and lattice parameters of sintered ceramics were identified by an X-ray diffraction pattern (XRD, D5000 Diffratometer, Siemens, Germany). The bulk densities of the sintered pellets were measured by the Archimedes method. The dielectric constant (ε_r) and the quality factor values ($Q \times f$) at microwave frequencies around 8.84 GHz were measured using the Hakki–Coleman dielectric resonator method which had been modified and improved by Courtney [17,18]. A system combined with an HP8757D network analyzer and an HP8350B sweep oscillator was employed in the measurement. The temperature coefficient of the resonant frequency (τ_f) value (ppm/°C) can be calculated

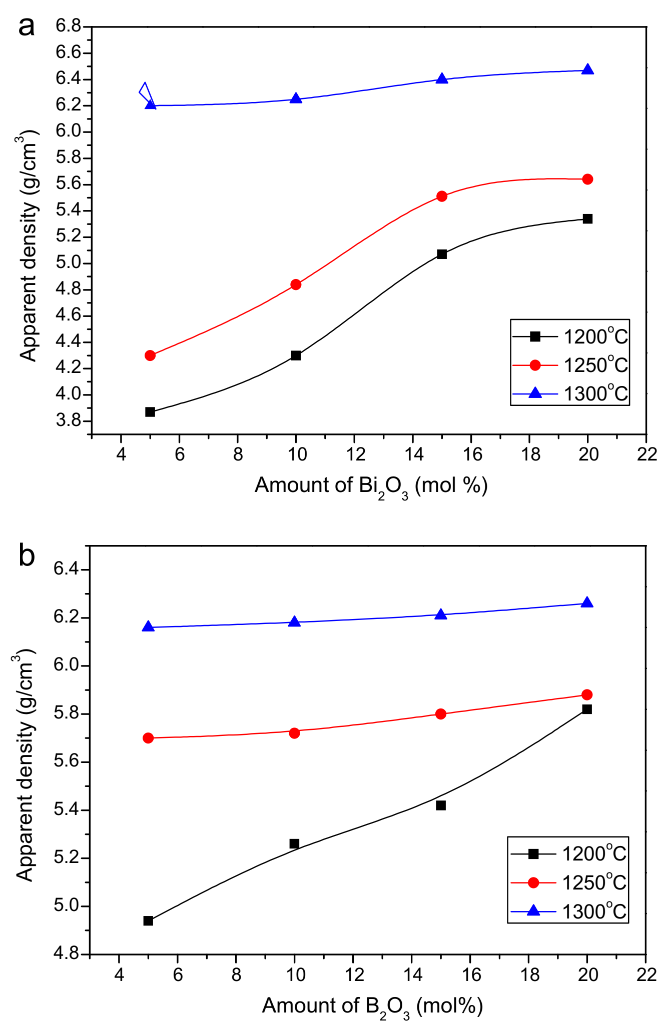

Fig. 1. The dependence of apparent density of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics with different mol% of (a) Bi_2O_3 and (b) B_2O_3 sintering aids.

by the change in resonant frequency (f),

$$\tau_f = \frac{f_2 - f_1}{f_1(T_2 - T_1)} \tag{1}$$

where f_1 and f_2 represent the resonant frequencies at temperatures $T_1 = 25\ ^\circ\mathrm{C}$ and $T_2 = 80\ ^\circ\mathrm{C}$, respectively.

3. Results and discussion

As shown in Fig. 1, it is found that $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics could not get satisfactory densities while sintering temperatures were less than 1300 °C, no matter what amounts of sintering aids are incorporated into $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics. If the sintering temperatures are higher than 1300 °C, we can obtain the better microwave dielectric properties. However, this behavior will disobey the aim of this research. Based on the above-mentioned reasons, we selected the sintering temperature at 1300 °C. The apparent densities increased with an increase in sintering temperature as well as the amount of sintering aids. Generally speaking, the densities of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with Bi_2O_3 are higher than those incorporated with B_2O_3. This is due to the fact that the molecular weight of Bi_2O_3 is larger than B_2O_3.

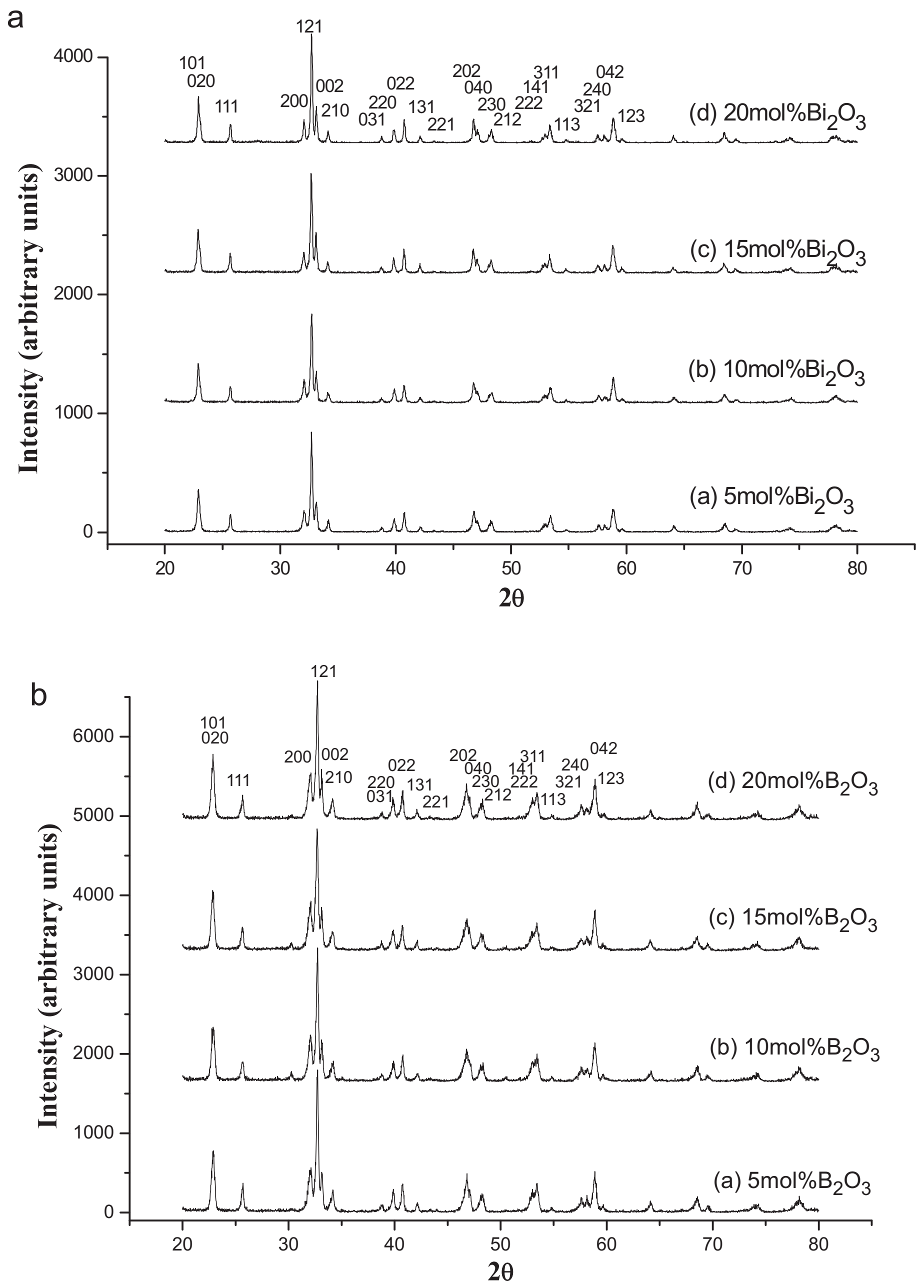

Fig. 2. (a) X-ray diffraction pattern of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics sintered at 1300 °C for 6 h with (a) 5 mol%, (b) 10 mol%, (c) 15 mol%, and (d) 20 mol% Bi_2O_3 additives. (b) X-ray diffraction pattern of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics sintered at 1300 °C for 6 h with (a) 5 mol%, (b) 10 mol%, (c) 15 mol%, and (d) 20 mol% B_2O_3 additives.

Table 1a
Lattice parameters, cell volumes, crystal symmetries of $Sm(Mg_{0.5}Ti_{0.5})O_3$ (SMT) ceramics for various Bi_2O_3 additions sintered at 1300 °C for 6 h.

Materials	Lattice parameter (nm)	Cell volume (nm^3)	Crystal symmetries
SMT + 5 mol% Bi_2O_3	$a = 0.5579$, $b = 0.7703$, $c = 0.5398$	0.2319	Monoclinic
SMT + 10 mol% Bi_2O_3	$a = 0.5582$, $b = 0.7718$, $c = 0.5401$	0.2326	Monoclinic
SMT + 15 mol% Bi_2O_3	$a = 0.5582$, $b = 0.7719$, $c = 0.5409$	0.2330	Monoclinic
SMT + 20 mol% Bi_2O_3	$a = 0.5586$, $b = 0.7723$, $c = 0.5.412$	0.2335	Monoclinic

Table 1b
Lattice parameters, cell volumes, crystal symmetries of $Sm(Mg_{0.5}Ti_{0.5})O_3$ (SMT) ceramics for various B_2O_3 additions sintered at 1300 °C for 6 h.

Materials	Lattice parameter (nm)	Cell volume (nm^3)	Crystal symmetries
SMT + 5 mol% B_2O_3	$a = 0.5568$, $b = 0.7694$, c = 0.5408	0.2316	Monoclinic
SMT + 10 mol% B_2O_3	$a = 0.5572$, $b = 0.7697$, $c = 0.5409$	0.2319	Monoclinic
SMT + 15 mol% B_2O_3	$a = 0.5575$, $b = 0.7701$, $c = 0.5419$	0.2326	Monoclinic
SMT + 20 mol% B_2O_3	$a = 0.5582$, $b = 0.7702$, $c = 0.5429$	0.2334	Monoclinic

Fig. 2(a) and (b) shows the X-ray diffraction patterns of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with different mol% of Bi_2O_3 and B_2O_3 sintered at 1300 °C for 6 h, respectively. All of the $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with various amounts of Bi_2O_3 and B_2O_3 exhibited perovskite phase with a monoclinic structure, which structure belongs to the $P2_1/b$ space group. To confirm the formation of the solid solution, lattice parameters, cell volumes, crystal symmetries of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with various amount of Bi_2O_3 and B_2O_3 additions sintered at 1300 °C for 6 h are

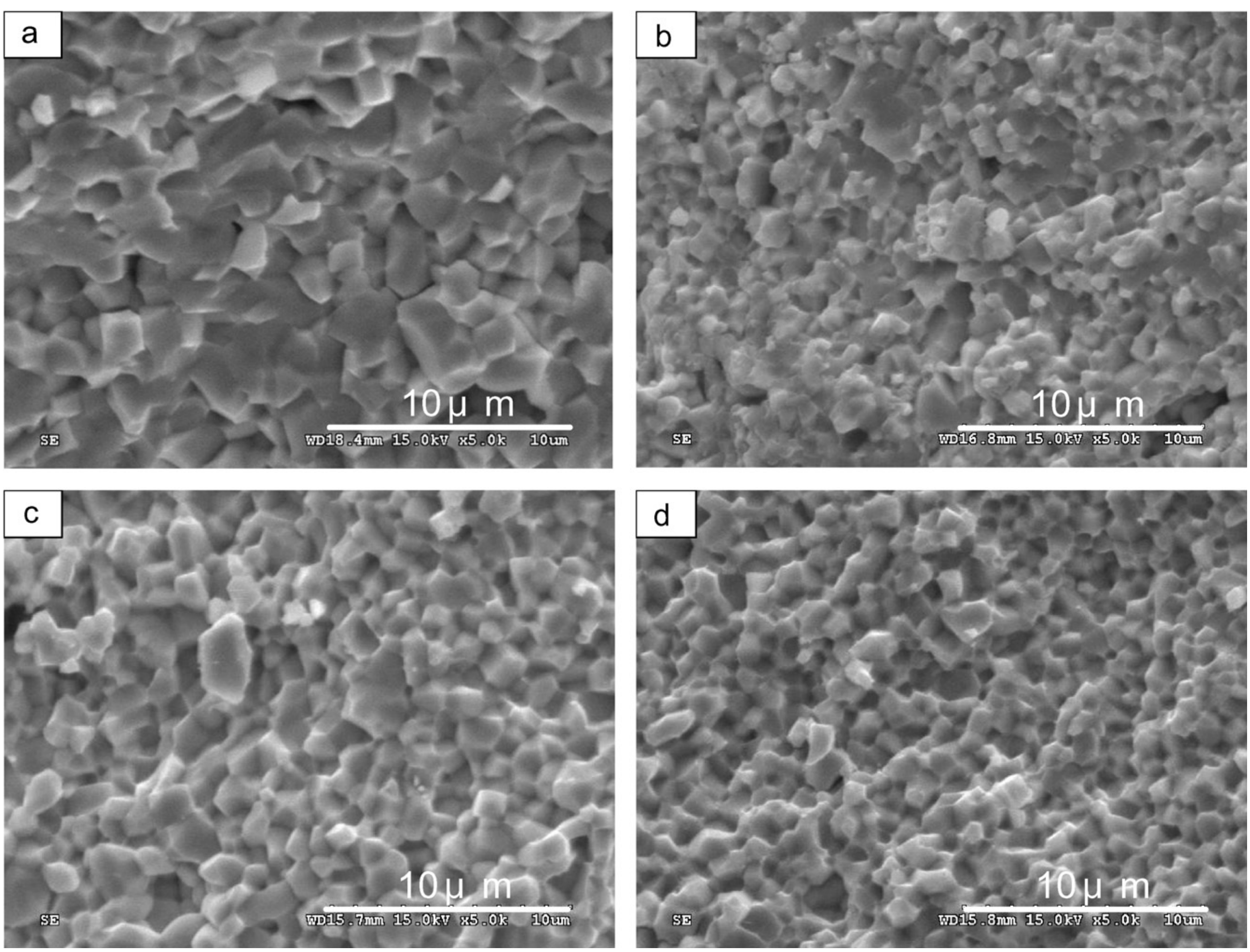

Fig. 3. The backscattered electron micrographs of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics sintered at 1300 °C for 6 h with (a) 5 mol%, (b) 10 mol%, (c) 15 mol% and (d) 20 mol% Bi_2O_3 additives.

listed in Tables 1a and 1b. It is found that the lattice parameter depends on the amount of sintering aids of Bi_2O_3 and B_2O_3 By doping sintering aids of Bi_2O_3 or B_2O_3 in $Sm(Mg_{0.5}Ti_{0.5})O_3$, lattice will induce uniform strain in the lattice as the material elastically deform. This effect causes the lattice plane spacing to change and the diffraction peaks to shift to new 2θ positions. For Bi_2O_3-doped specimens, the crystal symmetries are monoclinic. As increasing the amount of Bi_2O_3 addition, the lattice parameters (a, b, and c) and cell volumes are increased in which, the cell volumes are in the range of 0.2319–0.2335 nm^3 with various amount of Bi_2O_3. Similarly, B_2O_3-doped specimens, the crystal symmetries are also monoclinic. With increasing the amount of B_2O_3 addition, the lattice parameters (a, b, and c) and cell volumes are increased in which, the cell volumes are in the range of 0.2316–0.2334 nm^3 with various amount of B_2O_3. Because the ionic radius of Bi^{3+} for coordination number of 6 is 0.103 nm, whereas the ionic radius of B^{3+} is 0.027 nm clearly, the ionic radius of Bi^{3+} is greatly larger than that of B^{3+}. This results in the specimens incorporated with Bi_2O_3 having larger lattice parameters and cell volume than those incorporated with B_2O_3. The impurity phases or secondary phases were not detected in $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with sintering aids. It is due to the fact that the detection of the minor phase by XRD is extremely difficult.

Fig. 3 shows the images of fracture surface for $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with different mol% of Bi_2O_3, which indicates that the grain size decreased with the increasing amount of Bi_2O_3 addition. Fig. 4 shows the images of fracture surface for $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics sintered at 1300 °C incorporated with different mol% of B_2O_3. Depending on the interfacial tension, the liquid phase may penetrate the grain completely, in which case the grains will be separated from one another by a thin layer in $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with Bi_2O_3. Because these grain boundary thin films are very thin with a thickness of 0.5–2 nm between the grains, they were not easily found in the SEM images. Depending on the composition of the particulate solid and the liquid phase, a variety of grain shapes, ranging from nearly equiaxial grains to elongated grains are observed [19]. In $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with B_2O_3 case, the volume fraction of liquid is high, the grains may dissolve into a liquid phase, and rapidly rearrange, in which case contact points between agglomerates will be dissolved due to their

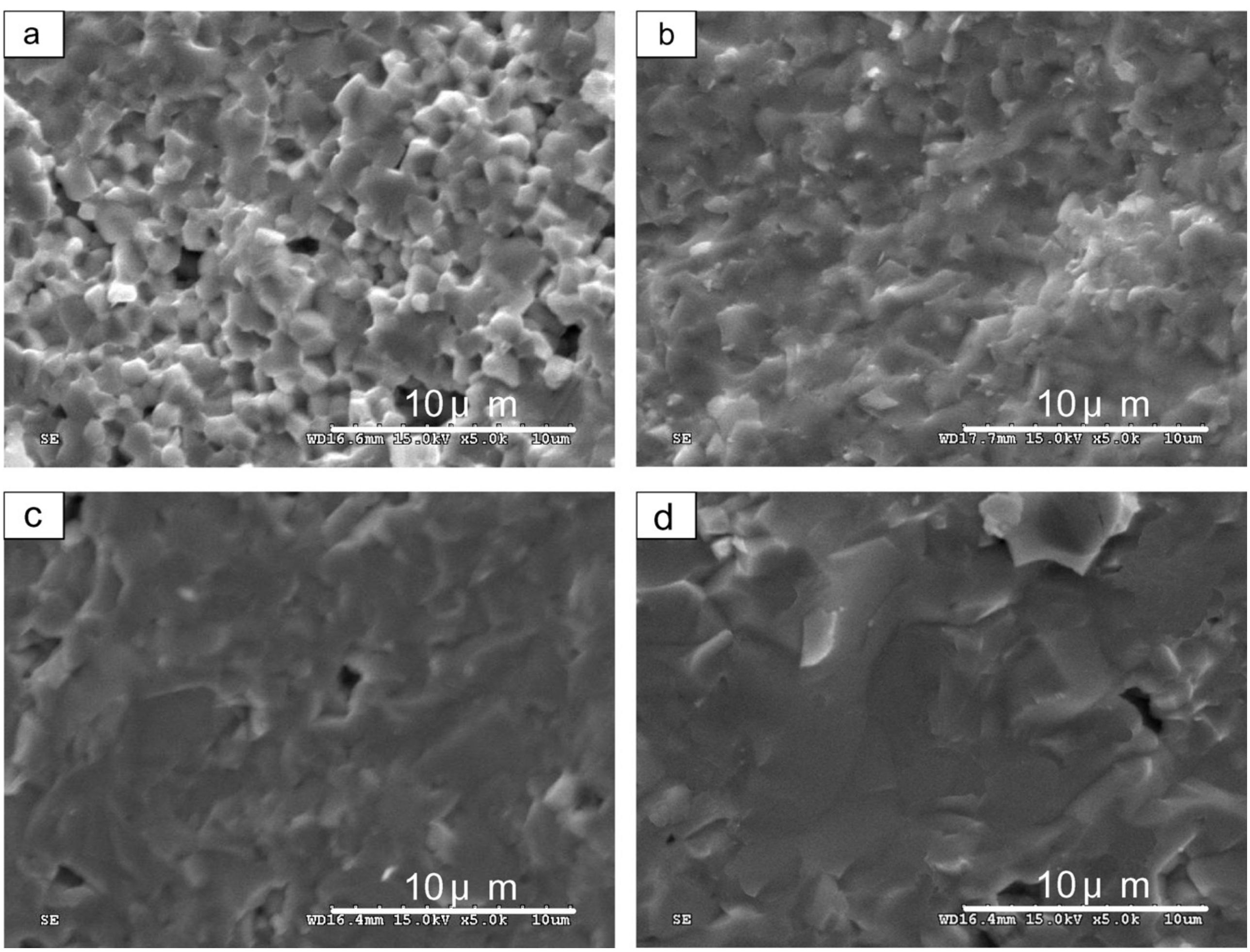

Fig. 4. The backscattered electron micrographs of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics sintered at 1300 °C for 6 h with (a) 5 mol%, (b) 10 mol%, (c) 15 mol% and (d) 20 mol% B_2O_3 additives.

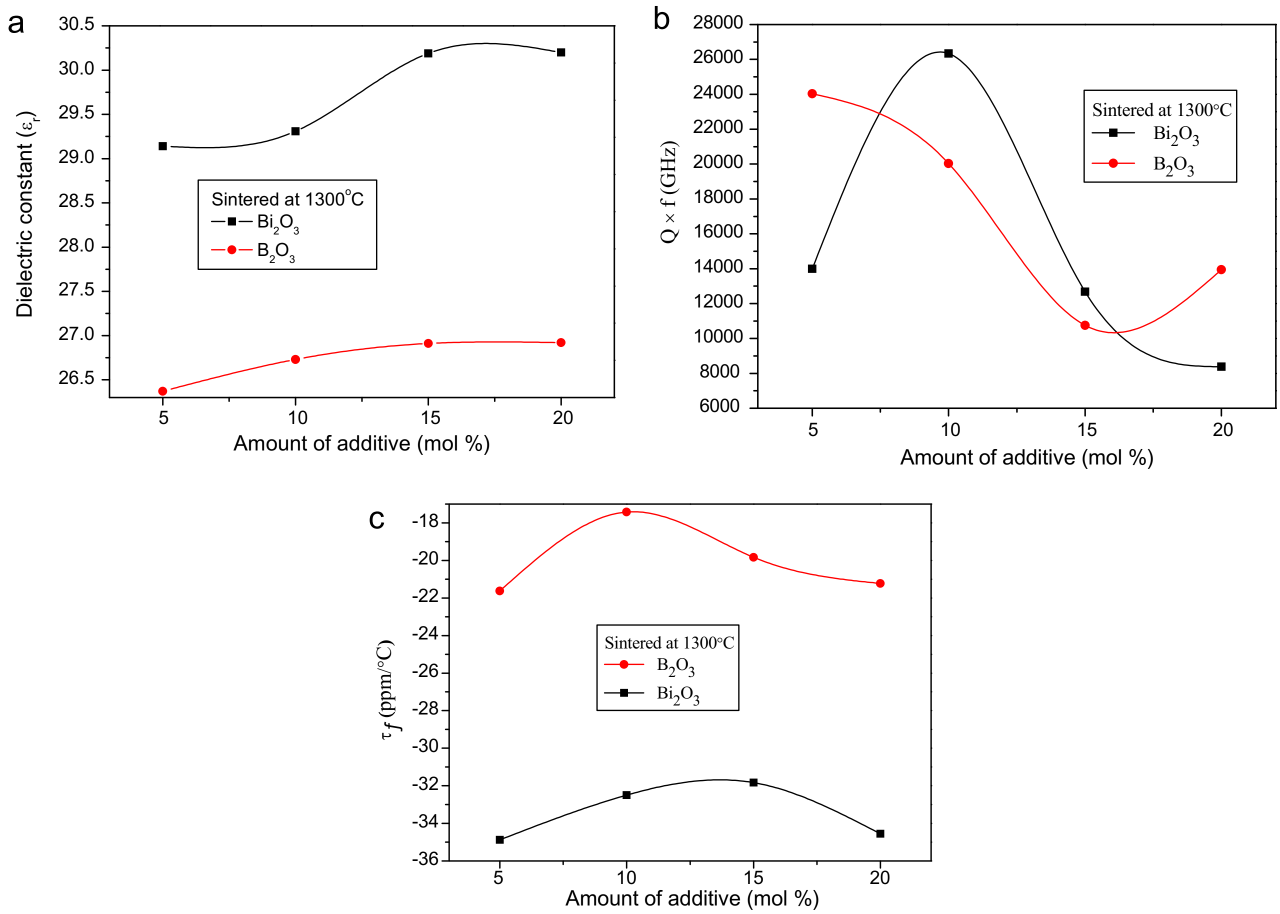

Fig. 5. (a) The dependence of dielectric constant of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics with different mol% of Bi_2O_3 and B_2O_3 additives. (b) The dependence of quality factor ($Q \times f$) of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics with different mol% of Bi_2O_3 and B_2O_3 additives. (c) The dependence of τ_f value of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics with different mol% of B_2O_3 and Bi_2O_3 additives.

higher solubility in the liquid. Throughout this process, dissolution of sharp edges will make the particle surface smoother, reducing the interfacial area and aiding the rearrangement of the system in which case, the microstructure will lead plate-like shape. With increasing the amount of B_2O_3, the area of plate-like particles increases as shown in Fig. 3(b)–(d). Therefore, the selection of a sintering aid is very important for microwave properties on microwave dielectric ceramics.

The dielectric constant (ε_r) of the $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics sintered at 1300 °C as function of incorporated amount of Bi_2O_3 and B_2O_3 is shown in Fig. 5(a). The dielectric constant of specimens was approximately proportional to the sintered bulk density of specimens. A maximum dielectric constant value of 30.2 was obtained for the specimen incorporated with 20 mol% Bi_2O_3 sintered at 1300 °C and the dielectric constant increases slightly with increasing B_2O_3 amount. Incorporated with 20 mol% of B_2O_3, a dielectric constant value of 26.8 was obtained for $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics. James Clerk Maxwell proposed that the dielectric constant of a random dispersion of spheres of the dielectric constant ε_1 in a matrix of relative dielectric constant ε_2 is given by

$$\varepsilon_m = \varepsilon_2\left\{1 + \frac{3V_f(\varepsilon_1 - \varepsilon_2)}{\varepsilon_1 + 2\varepsilon_2 - V_f(\varepsilon_1 - \varepsilon_2)}\right\}$$

where V_f is the volume fraction occupied by the dispersed particles. The result is independent of the size of the dispersed particles [20]. The dielectric constant of $Sm(Mg_{0.5}Ti_{0.5})O_3$ increases with increasing the amount of sintering aids. The value of dielectric constant of $Sm(Mg_{0.5}Ti_{0.5})O_3$ incorporated with Bi_2O_3 is larger than that of B_2O_3. This is due to the fact that the dielectric constant of Bi_2O_3 is larger than B_2O_3.

Fig. 5(b) shows the quality factor ($Q \times f$) of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics as a function of the incorporated amount of Bi_2O_3 and B_2O_3. The dielectric constant (ε_r) and the quality factor values ($Q \times f$) were measured at microwave frequencies around 8.84 GHz. For the Bi_2O_3 addition component, a maximum $Q \times f$ value of 26,000 GHz was obtained while incorporated with 10 mol% of Bi_2O_3. When the incorporated amount of Bi_2O_3 is larger than 10 mol%, the value of $Q \times f$ is gradually decreased. This is due to the fact that the uniform grain sizes appeared in incorporated with 10 mol% of Bi_2O_3. For the B_2O_3 addition component, a maximum $Q \times f$ value of 24,000 GHz was obtained for incorporated with 5 mol% of B_2O_3. With a further increase in the incorporated amount of B_2O_3, the $Q \times f$ value gradually decreased from 24,000 to 10,000 GHz as the incorporated amount of B_2O_3 increased from 5 mol% to 20 mol%. We concluded that (1) high amounts of Bi_2O_3 and B_2O_3 additions cause a liquid phase spread in $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics and degrades the $Q \times f$ value. (2) Large grain size usually brings high $Q \times f$ value due to the decrease of defect on the grain boundary. The reason for the degradation in the $Q \times f$ value should be the existence of grassy phase.

The temperature coefficients of resonant frequency (τ_f) of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics as a function of incorporated amount of Bi_2O_3 and B_2O_3 are illustrated in Fig. 5(c). The temperature coefficients of resonant frequency are well known, related to composition, the additives and the second phase of the material. For the B_2O_3 addition, the τ_f improved from −22 to −17.5 ppm/°C as the incorporated amount of B_2O_3 increased from 5 mol% to 10 mol% and decreased thereafter. For the Bi_2O_3 addition, the τ_f varied toward the positive direction with increasing incorporated amount of Bi_2O_3 from 5 mol% to 15 mol% and decreased thereafter. It implies that the τ_f value was not sensitive for sintering aids of B_2O_3 and Bi_2O_3 content because no significant secondary phase was observed.

4. Conclusion

The microwave dielectric properties of $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with different amount of Bi_2O_3 and B_2O_3 have been investigated systematically. In $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with B_2O_3 case, the volume fraction of liquid is high, the grains may dissolve into liquid phase, and rapidly rearrange, in which case the microstructure will form plate-like shape particles. The experimental results show that a dielectric constant (ε_r) of 29.3, a high $Q \times f$ value of 26,335 GHz (at 8.84 GHz), and a τ_f of −32.5 ppm/°C can be obtained for $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with 10 mol% Bi_2O_3 sintered at 1300 °C, while for $Sm(Mg_{0.5}Ti_{0.5})O_3$ ceramics incorporated with B_2O_3 case, the best result for B_2O_3 case is at 10 mol% B_2O_3, $\varepsilon_r = 26.8$, $Q \times f =$ 20,000 GHz, and $\tau_f = -17.5$ ppm/°C. These ceramics are suitable for miniaturization of the application of dielectric resonators and filters.

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