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Enhanced microwave dielectric properties of $Bi_6B_{10}O_{24}$ ceramics as ultra-low temperature co-fired ceramics materials

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ABSTRACT

The $Bi_6B_{10}O_{24}$ microwave dielectric ceramics for ultra-low temperature co-fired ceramics application were prepared by solid-state reaction method. The process conditions, phase composition, and microwave dielectric properties of the ceramics were investigated. The results indicated that boron volatilization was effectively avoided by dry ball milling of the raw materials and calcined in sealed environment for the powder preparation and then a single $Bi_6B_{10}O_{24}$ phase ceramic was successfully obtained. The $Bi_6B_{10}O_{24}$ ceramics exhibited microwave dielectric properties: $\varepsilon_r = 13.2 \pm 0.1$, $Q \times f = 25{,}000 \pm 200$ GHz, and $\tau_f = -65 \pm 2$ ppm/°C. The effect of small excess B_2O_3 on the phase, microstructure, and microwave dielectric properties on the $Bi_6B_{10}O_{24}$ ceramics were discussed. $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0$–0.12 mol%) were sintered at 670–710 °C for 2 h. The XRD patterns of specimens illustrated that only the $Bi_6B_{10}O_{24}$ phase was observed for all the ceramics with varying x. The microwave dielectric properties of the ceramics were found to strongly correlate with the x values and sintering process. The appropriate excess B_2O_3 is beneficial to densification of the ceramics, effectively reducing the dielectric loss and increasing $Q \times f$. The excellent microwave dielectric properties of the $Bi_6B_{10}O_{24}$ ceramics were obtained for $x = 0.1$ mol, $\varepsilon_r = 12.5 \pm 0.1$, $Q \times f = 38{,}200 \pm 300$ GHz, and $\tau_f = -62 \pm 1$ ppm/°C. The high-performance $Bi_6B_{10}O_{24}$ ceramics are promising candidates for ULTCC integration applications.

1 Introduction

With the rapid development of wireless communication, global positioning system (GPS), Internet of Things, and other information industries, the corresponding systems and devices are developing into miniaturization, multi-function, and low cost [1]. Low-temperature co-fired ceramic (LTCC) technology is an important way to realize the miniaturization, integration, and high reliability of components [2, 3]. In practical applications, excellent properties of microwave dielectric materials are required, for

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example, the appropriate dielectric constant (ε_r), high quality factor ($Q \times f$), and the near-zero temperature coefficient of the resonant frequency (τ_f). The research for LTCCs has accelerated in recent years, and ultra-low temperature co-fired ceramics (ULTCC) technology comes up and becomes one of hotspots. ULTCC requires lower sintering to co-firing with other low melting point electrode materials, which is beneficial to reduce energy consumption and improve the compatibility with economical and effective metal electrodes [4].

At current, a large number of excellent microwave dielectric materials have been reported in the literature; however, most of them have high sintering temperatures [5]. Usually, the sintering temperature can be reduced by adding low melting glass, but this method often cause the deterioration of microwave dielectric properties [6]. Therefore, some low melting oxides such as Li_2O-, TeO_2-, B_2O_3-, WO_3-, and MoO_3-based compounds with intrinsically low sintering temperature and promising microwave dielectric properties have been extensively studied [4, 7–9]. However, most of these materials have fetal defects, such as hygroscopic, unstable, poisonous, high cost and so on. As we known, B_2O_3-based microwave dielectric materials have stable chemical properties, and there are few researches on them at present. Ohashi et al. [10] prepared $Li_3AlB_2O_6$ ceramics by conventional solid-phase reaction method and obtained the microwave dielectric properties of $\varepsilon_r = 4.2$, $Q \times f = 13{,}027$ GHz, and $\tau_f = 10$ ppm/°C. CuO–B_2O_3–Li_2O (CBL) glass–ceramic [11] showed microwave dielectric properties with $\varepsilon_r = 5.84$, $Q \times f = 10{,}120$ GHz (at 13.44 GHz), and $\tau_f = -33$ ppm/°C.

Until now, borate ceramics with ultra-low sintering temperatures and excellent microwave dielectric properties are limited, so it is necessary to develop new borate ceramics. The B_2O_3–Bi_2O_3 system was a low melting point system, and previous researchers were focus on single crystal and glass materials for the optical properties [12–16]. The research on the ceramics was few. $Bi_4B_2O_9$ and $Bi_6B_{10}O_{24}$ ceramics in the B_2O_3–Bi_2O_3 system were firstly prepared as sintering aids by Chen et al. [17] through solid-state reaction and their microwave dielectric properties were reported, $\varepsilon_r = 39$, $Q \times f = 2600$ GHz, and $\tau_f = -203$ ppm/°C for $Bi_4B_2O_9$ ceramics and $\varepsilon_r = 10$, $Q \times f = 10{,}800$ GHz, and $\tau_f = -41$ ppm/°C for $Bi_6B_{10}O_{24}$ ceramics. Recently, Wang et al. [18] stoichiometrically weighted Bi_2O_3 and H_3BO_3 as raw powders to prepare $Bi_6B_{10}O_{24}$ ceramics, but the $Bi_4B_2O_9$ phase as second phase was appeared in the $Bi_6B_{10}O_{24}$ ceramics. The volatile of boron caused the second phase to occur in the ceramics. For this reason, excess boron (over 80% of the stoichiometric amount) was added to compensate for boron volatilization in order to obtain a single-phase $Bi_6B_{10}O_{24}$ ceramics. The microwave dielectric properties of the ceramics were $\varepsilon_r = 12.14$, $Q \times f = 14{,}800$ GHz, and $\tau_f = -72$ ppm/°C. It is obviously that microwave dielectric properties of $Bi_6B_{10}O_{24}$ ceramics are poor and a high amount of excess boron has to be added.

The $Bi_6B_{10}O_{24}$ ceramics exhibit low sintering temperature and are potential for ULTCCs. However, the properties obtained from the current researchers are not excellent enough. In this study, the massive volatilization of boron was avoided by dry ball milling and calcined in sealed environment for the powders preparation, making the actual composition of the ceramics to be consistent with the nominal composition. The effect of small excess B_2O_3 on the $Bi_6B_{10}O_{24}$ ceramics was discussed. The process conditions, microstructures, and microwave dielectric properties of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0$–0.12 mol%) as a microwave dielectric material for ultra-low temperature sintering are investigated.

2 Experimental

$Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0$–0.12 mol%) were prepared by the solid-state reaction route. Raw powders of B_2O_3 and Bi_2O_3 (AR grade, Sinopharm Chemical Reagent) were weighed as ratio of $Bi_6B_{10}O_{24}$–x B_2O_3 ($x = 0$–0.12 mol%). In the ball milling of raw materials, two ball milling methods were used for comparison, wet milling, and dry milling. The wet milling was ball milled for 8 h using agate balls with isopropanol as medium and dried at 85 °C for 8 h. The dry milling was ball milled for 2 h using agate balls without isopropanol as medium. After milling, both powders calcined at 660 °C for 2 h in sealed environment, respectively. The calcined powders were re-milled for 6 h with isopropanol as the dispersive media, dried at 85 °C for 8 h, and pressed into pellets (diameter 12 mm) using 5 wt% PVA as a binder. These pellets were heated at 400 °C for 10 h to eliminate the PVA and sintered at 660–710 °C for 2 h in air.

The crystalline phase of samples were identified by X-ray diffraction (XRD: PANalytical X'Pert PW3050/60 Philips, Netherlands) using Cu Kα (λ = 1.54056 Å) range of 10°–80° (at a speed of 0.2°/s). The microstructures of samples were observed using a scanning electron microscope (SEM: Zeiss Ultra Plus). The particle size was examined by the analysis of the images obtained using SEM. The FT-IR and Raman spectra were collected through Fourier transform infrared spectrometer (Thermo Nicolet) at room temperature. The bulk densities of the sintered pellets were measured by the Archimedes method. The microwave dielectric properties (the ε_r and the $Q \times f$ value) of samples were measured in the TE011 mode using a network analyzer (Agilent HPB8722ET; Agilent Technologies, Santa Clara, CA) and parallel boards. The temperature coefficient of resonant frequency

$$\tau_f = \frac{f_{85} - f_{25}}{60 \times f_{25}} \tag{1}$$

was experimentally measured in the temperature range of 25–85 °C.

3 Results and discussion

Figure 1a shows the XRD pattern of the Bi/B = 3/5 powder by wet ball milling calcined in the sealed environment. The main phase of the ceramic is the $Bi_4B_2O_9$ phase (PDF: 01-070-1458), and the $Bi_6B_{10}O_{24}$ (PDF: 01-070-0154) is indexed to the second phase. Figure 1b shows the XRD pattern of the Bi/B = 3/5 powder by dry ball milling calcined in the sealed environment, figuring that a single $Bi_6B_{10}O_{24}$ phase occurs without $Bi_4B_2O_9$ phase. The cell parameters of $Bi_6B_{10}O_{24}$ are a = 6.5320 Å, b = 7.7330 Å, c = 18.5660 Å, $\alpha = \beta = \gamma$ = 90°, and V = 937.80 Å^3 with orthorhombic structure in space group *Pnma* (62) (PDF: 01-070-0154). This result indicates that the volatilization of B_2O_3 occurs in the drying process after wet ball milling. It reveals that boron volatilization during drying for wet ball milling can effectively avoid by dry ball milling for powders preparation, making the actual composition of the powders to be consistent with the nominal composition, which is an important improvement on previous research results.

Figure 2 shows the XRD patterns for the Bi/B = 3/5 powders calcined for 2 h at 600–680 °C by dry ball milling. At 600–620 °C, $Bi_4B_2O_9$ and $Bi_6B_{10}O_{24}$ are observed. With the temperature increasing to 640–680 °C, all the peaks are matched with the $Bi_6B_{10}O_{24}$, suggesting the following reaction sequence:

$$2Bi_2O_3 + B_2O_3 \rightarrow Bi_4B_2O_9, \tag{2}$$

$$3Bi_4B_2O_9 + 7B_2O_3 \rightarrow 2Bi_6B_{10}O_{24}. \tag{3}$$

It reveals that the pure $Bi_6B_{10}O_{24}$ phase powders can be prepared at suitable calcined temperature. The calcined temperature of powders in this study is chosen at 660 °C.

Figure 3 shows the relative density and microwave dielectric properties of the $Bi_6B_{10}O_{24}$ ceramics at different sintering temperatures. The ε_r and τ_f are basically stable as the sintering temperature changes, because all samples are with pure $Bi_6B_{10}O_{24}$ phase and high density. The $Q \times f$ reaches maximum of 25,000 ± 200 GHz at 660–670 °C and decreases significantly as sintering temperature further increasing, indicating that the microwave properties of the ceramics are affected by densification and sintering temperature. As the sintering temperature increases, it is gradually approaching the melting point of $Bi_6B_{10}O_{24}$, resulting in the appearance of excess glass content, which deteriorates microwave dielectric properties of the ceramics.

By improving the powders preparation processes, i.e., dry ball milling and calcining in sealed environment, single $Bi_6B_{10}O_{24}$ phase ceramics sintered at 660–670 °C are obtained in the study: ε_r = 13.2 ± 0.1, $Q \times f$ = 25,000 ± 200 GHz, and τ_f = − 65 ± 2 ppm/°C. In the previous research of the low melting point B_2O_3–Bi_2O_3 system [18], a high amount of excess B_2O_3 affects the phase composition, microstructure, and microwave dielectric properties of the ceramics. In this work, the low melting point and volatility of B_2O_3 may cause a very small amount of volatilization during the preparation process even if above improved powders preparation processes are adopted to obtain the pure $Bi_6B_{10}O_{24}$ ceramics. Therefore, a small amount of extra B_2O_3 is added to compensate for possible volatilization and as sintering additive to promote sintering process.

The XRD patterns for $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics (x = 0–0.12 mol%) sintered at 700 °C for 2 h in sealed environment are shown in Fig. 4. All the peaks of the ceramics with the various boron contents are indexed

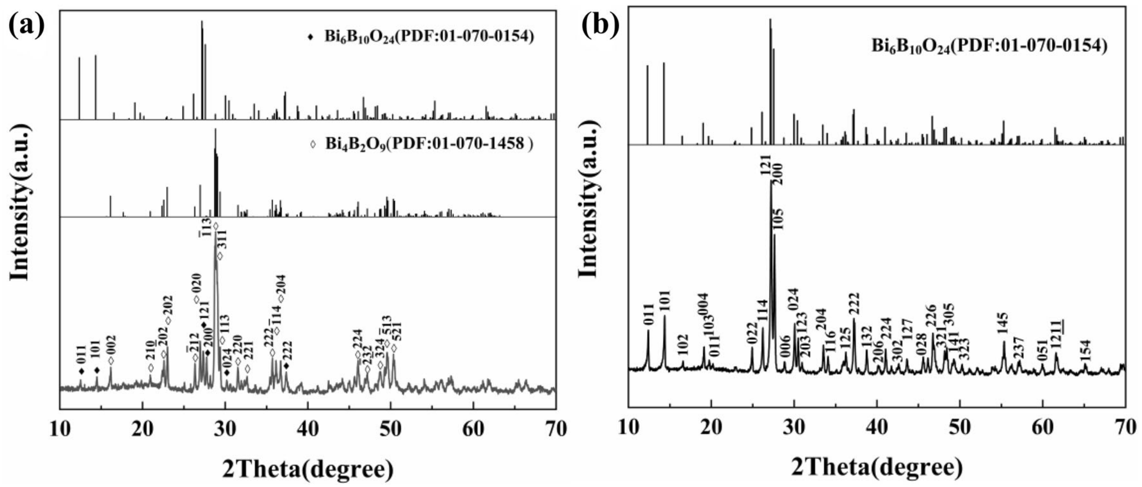


Fig. 1 The XRD patterns of the Bi/B = 3/5 powders calcined at 660°C for 2 h by different mixture methods. **a** Wet ball milling and **b** dry ball milling

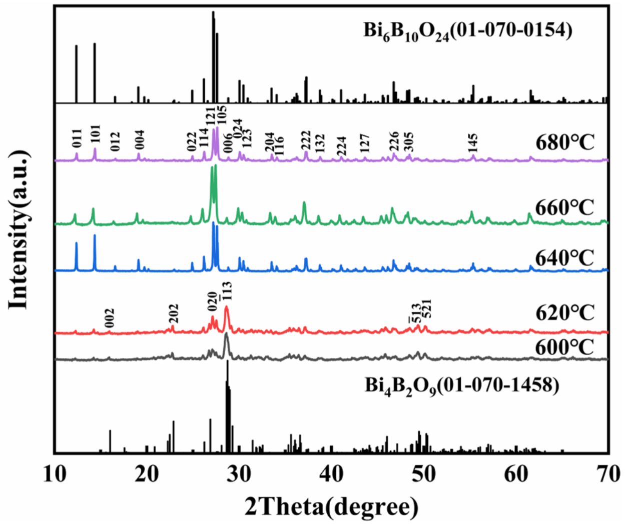


Fig. 2 The XRD patterns of the Bi/B = 3/5 powders calcined for 2 h at various temperatures by dry ball milling

as $Bi_6B_{10}O_{24}$ phase, and no other phases are detected. It reveals that small excess B_2O_3 does not change the phase composition of the ceramics.

$Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0$, 0.1 mol%) were also characterized by FT-IR and Raman spectroscopy. Figure 5 shows the FT-IR absorption spectra and Raman spectra of the $Bi_6B_{10}O_{24}$ ceramics sintered at 700 °C, respectively. The peaks of both samples are compared with the characteristic peaks of the known materials in Bi_2O_3–B_2O_3 system [19, 20]. All the peaks in the FT-IR and Raman spectrum of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0$, 0.1 mol%) are well matched with previous reports, and all agree well with the groups of the $Bi_6B_{10}O_{24}$ phase. It can be considered that only the $Bi_6B_{10}O_{24}$ phase is formed in both ceramics. These results also agree well with the above XRD analysis.

Figure 6 shows the variations of sintering temperature, ε_r, $Q \times f$, and τ_f of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0$–0.12 mol%). As x increases from 0 to 0.12, the variations of the ε_r and τ_f are not significant. The optimal sintering temperature of specimens increases. The $Q \times f$ increases with increasing x values, reaches a maximum of 38,200 ± 300 GHz for $x = 0.1$, and then decreases when $x = 0.12$. The ε_r and τ_f are mainly determined by the phase composition. According to the above XRD analysis, there is no secondary phase in all the samples. The phase composition of the ceramics is only $Bi_6B_{10}O_{24}$, therefore the ε_r and τ_f values of the ceramics have little change and are about 12.5 ± 0.1 and -62 ± 1 ppm/°C, respectively. The $Q \times f$ depends on extrinsic and intrinsic factor. The intrinsic losses are associated with lattice vibration modes while the extrinsic losses are associated with density, second phase, and so on [21, 22]. Therefore, higher density means lower porosity and higher $Q \times f$ value. When $x = 0$, the average grain size of the ceramic is 4.2 μm. With the x increases from 0.03 to 0.1, the average grain sizes of the samples increased from 2.1 to 3.6 μm, as shown in Fig. 7a–e. Obviously, their average grain sizes are smaller than the size of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0$). The grain size of the ceramics decreased with the content of glass increase in it [23]. The excess of B_2O_3 (x) results in the increase the glass in the ceramics. The average grain size is obviously affected by the x and sintering temperature. The appropriate

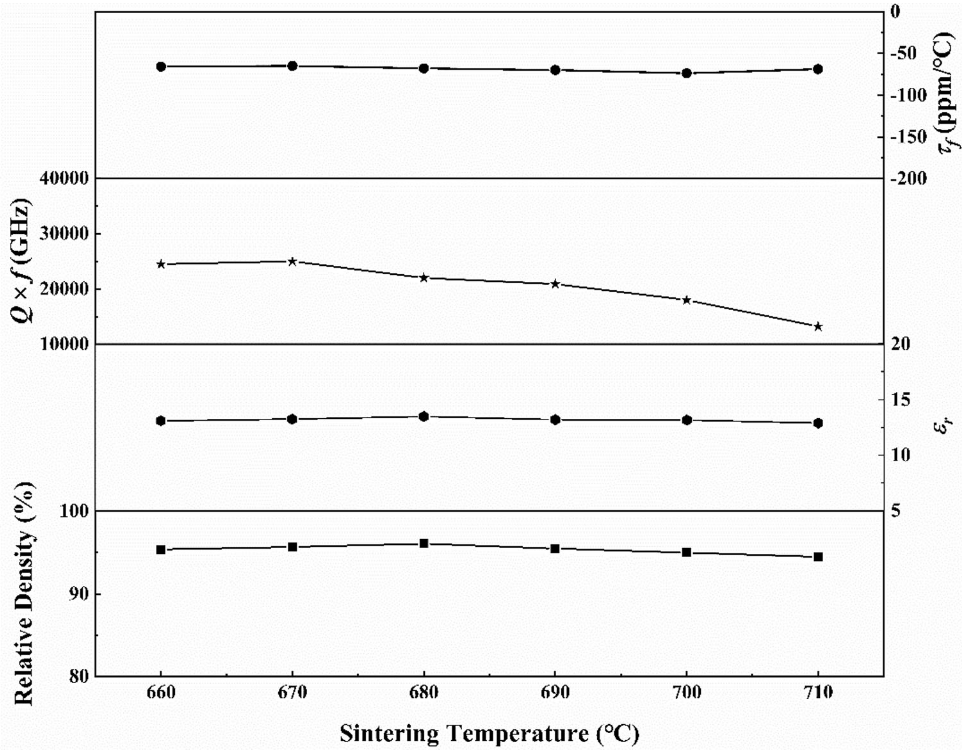


Fig. 3 Relative density, ε_r, $Q \times f$, and τ_f of $Bi_6B_{10}O_{24}$ ceramics at different sintering temperatures

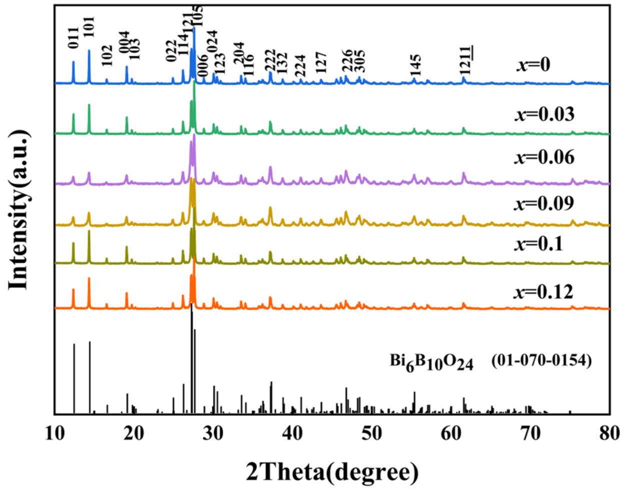


Fig. 4 The XRD patterns of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics (x = 0–0.12 mol%) sintered at 700°C for 2 h

excess B_2O_3 makes porosity decreases and densification of the ceramic at the optimum sintering temperature. The best microwave dielectric properties are obtained when $x = 0.1$, $\varepsilon_r = 12.5 \pm 0.1$, $Q \times f = 38{,}200 \pm 300$ GHz, and $\tau_f = -62 \pm 1$ ppm/°C. This phenomenon is mainly due to the liquid phase formed by the excess B_2O_3, which reduces the porosity of the ceramic, promotes the release of stress, and makes the ceramic sintered densely, thereby reducing the dielectric loss and increasing $Q \times f$. Almost all the samples could be densified at their optimal sintering temperature. At $x = 0.12$, more glasses exist, resulting in the grain size decrease with average size of 1.6 μm and the porosity increase (Fig. 7f) and the $Q \times f$ decrease. The more extra B_2O_3 deteriorates the microwave dielectric properties of the ceramics. It illustrates that an appropriate excess B_2O_3 promotes the densification of ceramics and improves their microwave dielectric properties.

The relative density and microwave dielectric properties of ceramics ($x = 0.1$) at different sintering temperatures are exhibited in Fig. 8. It shows that the microwave dielectric properties are greatly affected by the sintering temperature. The ceramics can be densified at 700–710 °C, and the microwave dielectric properties is significantly improved. With the sintering temperature from 660 to 710 °C, the average grain sizes increase (Fig. 9a–f), and the average grain sizes are 1.5, 1.5, 1.8, 2.5, 2.5, and 3.6 μm, respectively. The best microwave dielectric properties are obtained for 2 h, $\varepsilon_r = 12.5 \pm 0.1$, $Q \times f = 38{,}200 \pm 300$ GHz, and $\tau_f = -62 \pm 1$ ppm/°C.

The microwave dielectric properties of the ceramics studied in the Bi_2O_3–B_2O_3 system are listed in Table 1. In the previous studies of Bi_2O_3–B_2O_3 system, $Bi_6B_{10}O_{24}$ has better microwave dielectric

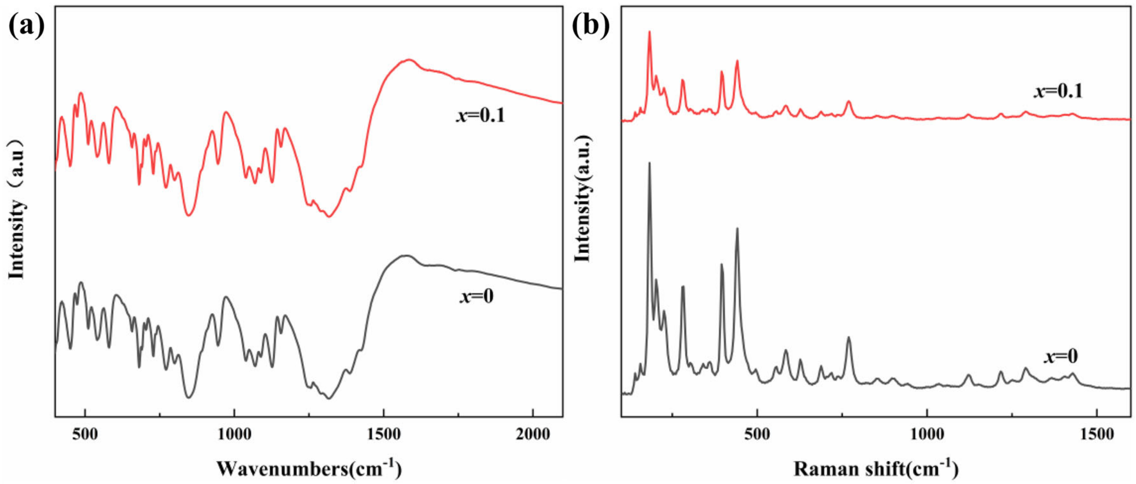
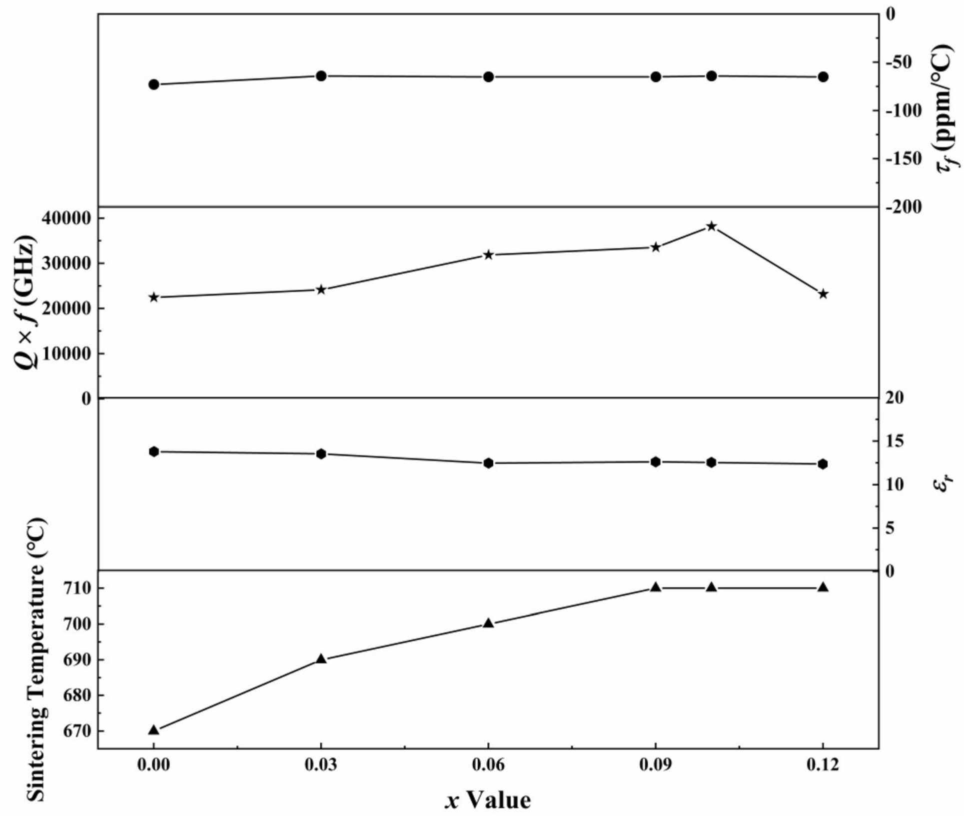


Fig. 5 **a** FT-IR absorption spectra and **b** Raman spectra of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics (x = 0, 0.1 mol%) sintered at 700°C for 2 h

Fig. 6 Sintering temperature, ε_r, $Q \times f$, and τ_f of the $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics (x = 0–0.12 mol%)



properties. However, previous studies have failed to obtain excellent properties of the pure $Bi_6B_{10}O_{24}$ ceramic which limit their applications. The enhanced microwave dielectric properties of the ceramics with pure $Bi_6B_{10}O_{24}$ phase are obtained in this study, with an ε_r of 12.5 ± 0.1, a high $Q \times f$ of 38,200 ± 300 GHz, and a τ_f of − 62 ± 1 ppm/°C, and the consistency of the raw material ratio and the target $Bi_6B_{10}O_{24}$ phase are guaranteed in our work, showing the potential of the $Bi_6B_{10}O_{24}$ ceramics in the field of excellent ULTCC microwave dielectric materials.

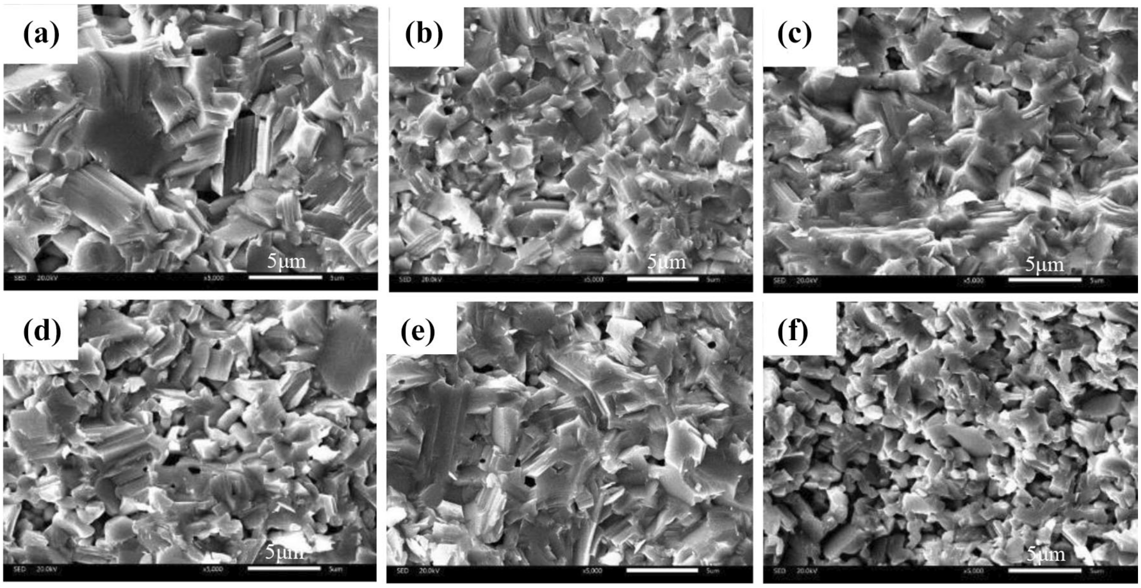
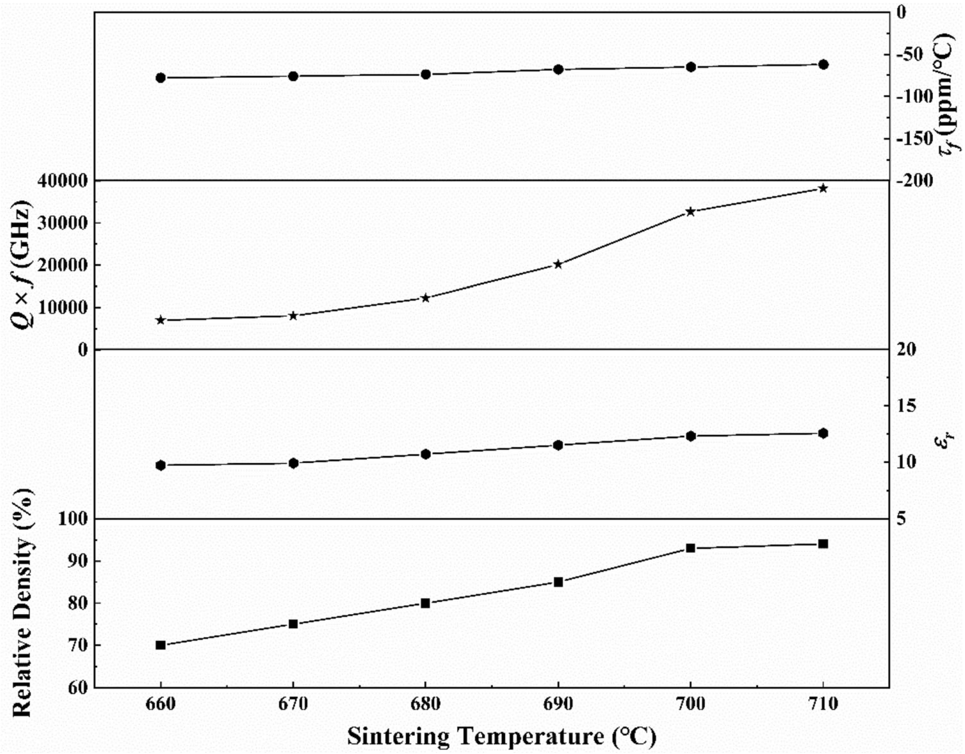


Fig. 7 SEM images of fracture surface of the $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics (x = 0–0.12 mol%) sintered at the optimal temperature. **a** $x = 0$, **b** $x = 0.03$, **c** $x = 0.06$, **d** $x = 0.09$, **e** $x = 0.1$, and **f** $x = 0.12$

Fig. 8 Relative density, ε_r, $Q \times f$, and τ_f of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics ($x = 0.1$ mol%) as a function of sintering temperature



4 Conclusion

$Bi_6B_{10}O_{24}$ ceramics in the Bi_2O_3–B_2O_3 system with high $Q \times f$ were investigated. The boron volatilization was effectively avoided by dry ball milling of the raw materials and calcined in sealed environment for the powder preparation, and a single $Bi_6B_{10}O_{24}$ phase ceramic was successfully obtained. Comparing with previous work, the $Bi_6B_{10}O_{24}$ ceramics exhibited enhanced microwave dielectric properties of $\varepsilon_r = 13.2 \pm 0.1$, $Q \times f = 25{,}000 \pm 200$ GHz, and $\tau_f = -65 \pm 2$ ppm/°C. The effect of small excess

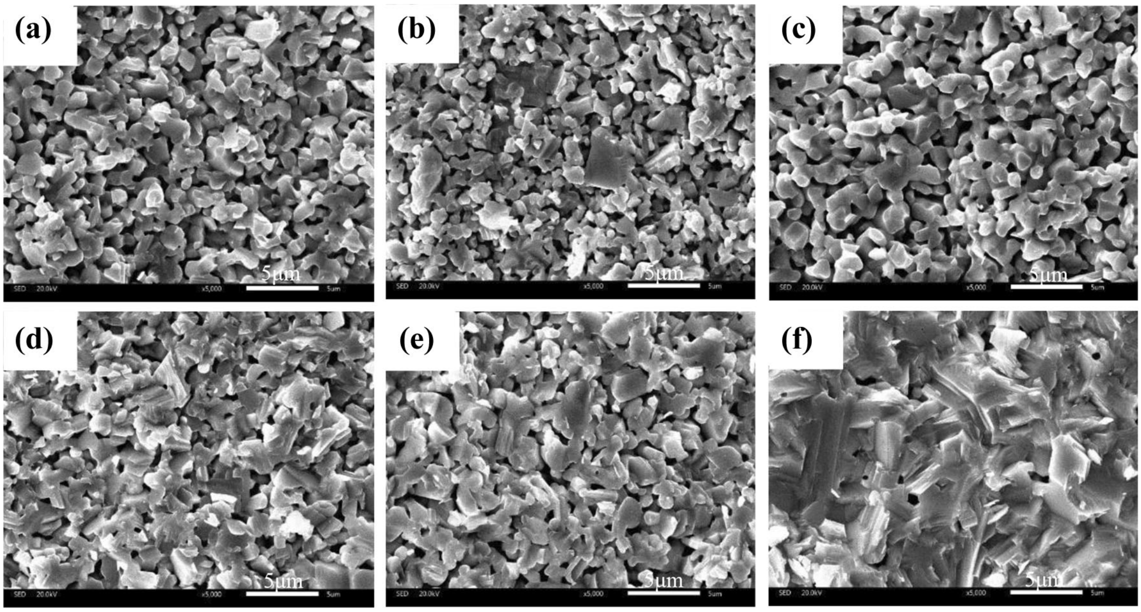


Fig. 9 SEM images of fracture surface of $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics (x = 0.1 mol%) sintering at **a** 660 °C, **b** 670 °C, **c** 680 °C, **d** 690 °C, **e** 700 °C, and **f** 710 °C for 2 h

Table 1 Comparison of the microwave dielectric properties of the ceramics in the Bi_2O_3–B_2O_3 system

Material	Phase	ε_r	$Q \times f$	τ_f	References
$Bi_4B_2O_9$	$Bi_4B_2O_9$	39	2600	− 203	Chen et al. [17]
$Bi_6B_{10}O_{24}$	$Bi_6B_{10}O_{24}$	10	10,800	− 41	Chen et al. [17]
$Bi_3B_{5+x}O_{12+3x/2}$ (x = 0)	$Bi_6B_{10}O_{24}$ + $Bi_4B_2O_9$	10.4	4600	− 100	Wang et al. [18]
$Bi_3B_{5+x}O_{12+3x/2}$ (x = 4)	$Bi_6B_{10}O_{24}$	12.1	14,800	− 72	Wang et al. [18]
$Bi_6B_{10}O_{24}$–xB_2O_3 (x = 0)	$Bi_6B_{10}O_{24}$	13.2	25,000	− 65	This work
$Bi_6B_{10}O_{24}$–xB_2O_3 (x = 0.1)	$Bi_6B_{10}O_{24}$	12.5	38,200	− 62	This work
Bi_2O_3	Bi_2O_3	33.5	18,700	− 235	Zhou et al. [24]

B_2O_3 on the $Bi_6B_{10}O_{24}$–xB_2O_3 ceramics (x = 0–0.12 mol%) was discussed. As x changed from 0 to 0.12, the excess B_2O_3 does not change the phase composition of the ceramics, while the microwave dielectric properties of $Bi_6B_{10}O_{24}$ ceramics changed significantly. Appropriate excess B_2O_3 promotes the densification of ceramics and improves their microwave dielectric properties. When x = 0.1, sintered at 710 °C for 2 h, $Bi_6B_{10}O_{24}$ ceramics possess excellent microwave dielectric properties of $\varepsilon_r = 12.5 \pm 0.1$, $Q \times f = 38{,}200 \pm 300$ GHz, and $\tau_f = -62 \pm 1$ ppm/°C. The ultra-low sintering temperature and excellent properties reveal the potential of $Bi_6B_{10}O_{24}$ ceramics for applications as a new ULTCC material.

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Author contributions

SS and QZ contributed to all the experimental work, completed the data analysis, and wrote the manuscript. YD guided all the experimental design and led the manuscript revision work. XP approved the final manuscript.

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Data availability

All data analyzed are used to support this study and are included in the submitted article.

Declarations

Conflict of interest The authors declared that they have no competing financial interests.

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