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Low-temperature sintering and microwave dielectric properties of Al_2TeO_6–TeO_2 ceramics

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ABSTRACT

We propose Al_2TeO_6–TeO_2 ceramics as a candidate for use as low-temperature co-fired ceramics (LTCC). We investigated microwave dielectric properties and low-temperature sintering conditions for Al_2TeO_6–TeO_2 ceramics. The calcined Al_2TeO_6 powders were sintered at 900 °C for 2–10 h with 30–50 wt% additive TeO_2. X-ray powder diffraction patterns showed that the sintered samples were Al_2TeO_6–TeO_2 composite with no other phase. The apparent density was improved with the additive TeO_2 content of up to 45 wt%. The dielectric constant (ε_r) increased by adding TeO_2 content from 35 to 45 wt%, although the quality factor ($Q \cdot f$) decreased. During sintering at 900 °C, the ε_r of the Al_2TeO_6–TeO_2 decreased slightly, whereas the $Q \cdot f$ increased gradually. The observed microstructures showed that the longer sintering time makes fewer pores in Al_2TeO_6–TeO_2 ceramics. Sintering at 900 °C for a long time contributes to densification, but it simultaneously causes TeO_2 evaporation. To prevent TeO_2 evaporation, we investigated the effects of annealing at 750 °C after sintering at 900 °C. Apparent densities or ε_r for the annealed samples were higher than those of the non-annealed samples. The $Q \cdot f$ improved with increasing annealing duration time, suggesting that sintering proceeded well during annealing with slower TeO_2 evaporation at 750 °C. The results show that annealing at 750 °C is effective to facilitate sintering and to control TeO_2 evaporation.

1. Introduction

Recently, low-temperature co-fired ceramics (LTCC) have attracted attention for the development of next-generation miniaturized microwave/millimeterwave communication devices [1–10]. Using the LTCC, co-firing of dielectric ceramics with internal electrodes is possible, where silver is usually used for the electrode. Because the melting point of silver is approximately 961 °C in air [11], the sintering temperature for the dielectric ceramics must be lower than 961 °C. However, until now, almost all micro/millimeter-wave dielectric ceramics require sintering temperatures higher than 1000 °C to obtain a higher quality factor ($Q \cdot f$), resulting from good densification and crystallinity [2,12–16]. Higher $Q \cdot f$ is an important factor for higher-frequency telecommuting applications such as filters and resonators [2]. This study was conducted to develop new dielectric ceramics with a high quality factor, even though the sintering temperature of the ceramics is lower than 961 °C of the silver melting point.

We have proposed that ceramics based on Al_2TeO_6 are candidate compounds for the LTCC because they contain Te oxide, which is characterized by its low melting point. For example, the melting point of TeO_2 is approximately 730 °C [17]. Some studies have been reported that the Te based binaries such as TiO_2–TeO_2, Bi_2O_3–TeO_2, and CuO–TeO_2 can be densified by sintering at temperatures lower than 900 °C [3–6,10]. Additionally, the Al based oxides tend to show lower dielectric constant [2,13,15,16]. Lower dielectric constant in addition to lower dielectric loss is also important for miniaturization of the higher-frequency telecommuting devises [2]. Su et al. have described that the Al_2TeO_6 possesses both of low-dielectric constant (ε_r) and dielectric loss ($\tan\delta$) in the frequency range of 1 kHz–1 MHz [18]. The ε_r and the $\tan\delta$ at 1 MHz have been reported as 9.4 and 0.03, respectively. That fact implies that the Al_2TeO_6 is also characterized by low-dielectric constant and dielectric loss in the microwave frequency range. However, no reports in the relevant literature describe studies of microwave dielectric properties in the Al_2TeO_6.

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Given that perspective, we tried earlier to prepare Al_2TeO_6 ceramic samples as a pilot study, but it was difficult to obtain dense ceramics, when the sample was sintered at temperatures below 960 °C. Not sintered sample, but the pressed sample was used to investigate the dielectric properties in the frequency range of 1 kHz–1 MHz in the literature [18]. It also presumes that preparing the sintered Al_2TeO_6 is difficult, because dielectric properties are usually investigated by using a sintered sample. Particularly, densification is more significant for the quality factor in the microwave frequency range. To overcome the difficulty, this study was undertaken to prepare dense Al_2TeO_6 ceramics by adding TeO_2 with the lower melting point. We investigated the low-sintering condition and crystallinity of Al_2TeO_6–TeO_2 ceramics. Then we investigated the microwave dielectric properties of the prepared samples and discussed relation between the sintering conditions and the microwave dielectric properties.

2. Experimental procedures

Al_2O_3 (purity: 99.99%) and TeO_2 (purity: 99.9%) were weighed as the mole ratio of 1:1. They were ball-milled for 24 h in ethanol solvent using alumina balls with 5 mmϕ diameter. After drying, the mixed powders were calcined in air at 550–650 °C for 10 h. The calcined Al_2O_3–TeO_2 powders were mixed with additional TeO_2 of 30–50 wt% and were again ball-milled for 24 h. The powders were molded into pellets with uniaxial pressure of approximately 7.8 MPa. Then cold isostatic pressing (CIP) of the molded samples was conducted under pressure of 200 MPa. The obtained pellets were sintered at 900 °C for 2–10 h in air. Some pellets were annealed at 750 °C for 24 h after sintering at 900 °C for 2 h.

The crystalline phase of the prepared sample was characterized at room temperature using X-ray powder diffraction (XRPD; X'pert MPD Pro; PANalytical B.V.) with Cu Kα radiation, where the conditions of XRPD voltage and current were respectively 45 kV and 40 mA. The apparent densities of the prepared samples were measured using Archimedes' method. The microstructure images of the prepared samples were investigated using a scanning electron microscope (SEM; JSM-6330F; JEOL Inc.). Differential thermal analysis (DTA) and thermogravimetry (TG) were conducted at temperatures of 20–900 °C with a rate of 10 °C/min to investigate the weight change and the melting point of TeO_2 in the samples.

The microwave dielectric properties of the prepared samples were investigated using the Hakki and Coleman method [2,19], for which a pellet sample was positioned between two copper plates. The microwave signal from the sample was investigated using a network analyzer (8720ES; Agilent Technologies Inc.). The dielectric constant (ε_r) and the quality factor ($Q \cdot f$) were calculated using the TE_{011} resonant mode investigated at room temperature [2,19]. The temperature coefficient of the resonator frequency (τ_f) was investigated at temperatures of 20–80 °C.

3. Results and discussion

3.1. Calcination condition to synthesize Al_2TeO_6

XRPD patterns of the Al_2O_3–TeO_2 powders calcined at 550–650 °C for 10 h are presented in Fig. 1. When the calcination temperature was 550 °C, reflections corresponding to Al_2O_3 and tetragonal TeO_2 were observed. At the calcination temperature of 600 °C, reflections corresponding to Al_2TeO_6 appeared in addition to the Al_2O_3 and the TeO_2 reflections. With higher calcination temperatures, the reflection intensities of the TeO_2 and the Al_2O_3 decreased, whereas the Al_2TeO_6 reflection intensities increased. When the calcination temperature was 620 °C, only Al_2TeO_6 reflections were observed, indicating that single phase of Al_2TeO_6 is obtainable at this calcination temperature. The reported TGA has showed an endothermal peak related to the formation of Al_2TeO_6 at 620 °C [18], which is good agreement with this XRPD result. We confirmed that a broad XRPD peak was observed around $2\theta = 25°$, coming from the presence of TeO_2 glass, when the Al_2O_3–TeO_2 powders were sintered at 650 °C. Consequently, we concluded that 620 °C is an appropriate condition for the calcination temperature for Al_2TeO_6.

3.2. Sintering condition to prepare dense Al_2TeO_6–TeO_2 ceramic samples

XRPD patterns of the sintered Al_2TeO_6–TeO_2 samples are portrayed in Fig. 2, where the samples were prepared by sintering the calcined Al_2TeO_6 powders with additive 30–50 wt% TeO_2 at 900 °C for 2 h. The calcined temperature was 620 °C, as described above. The observed reflections of the sintered samples can be indexed by two phases: Al_2TeO_6 and tetragonal TeO_2. With increasing additive TeO_2 contents, the TeO_2 reflection intensities increased slightly.

XRPD patterns of the Al_2TeO_6 with additive 40 wt% TeO_2 sintered at 900 °C for 2–10 h are depicted in Fig. 3. Reflections corresponding to Al_2TeO_6 and tetragonal TeO_2 were observed. With longer sintering duration time, the reflection intensities of the TeO_2 decreased gradually, suggesting that the TeO_2 is evaporated during sintering at 900 °C.

TG and DTA of the Al_2TeO_6 with additive 40 wt% TeO_2 are presented in Fig. 4. In the DTA, the endothermic peak was observed at 707 °C, at which point the sample weight began to decrease. The results demonstrate that TeO_2 contained in the sample begins to melt at 707 °C.

The relations between apparent densities and the sample preparation conditions ((1) additive TeO_2 amount and (2) sintering duration time) are presented in Fig. 5. It is difficult to estimate the relative densities in the Al_2TeO_6–TeO_2 composite series because TeO_2 is evaporated during sintering. In the case of the additive 30 wt% TeO_2, the apparent density was the lowest among the samples in the case of additive TeO_2 of 30–50 wt%, when the sintering duration time was 2 h. With increasing the additive TeO_2 from 30 wt% to 45 wt%, the apparent density increased. The

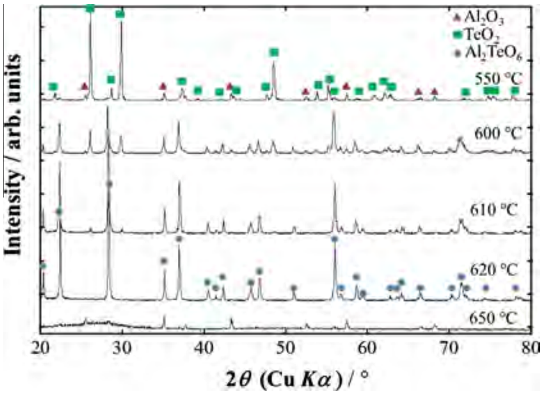

Fig. 1. XRPD patterns of the Al_2O_3–TeO_2 calcined for 10 h at 550–650 °C.

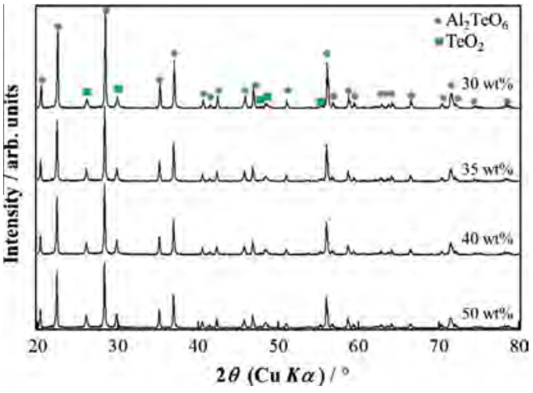

Fig. 2. XRPD patterns of the Al_2TeO_6 sintered for 2 h with additive TeO_2 of 30–50 wt% at 900 °C.

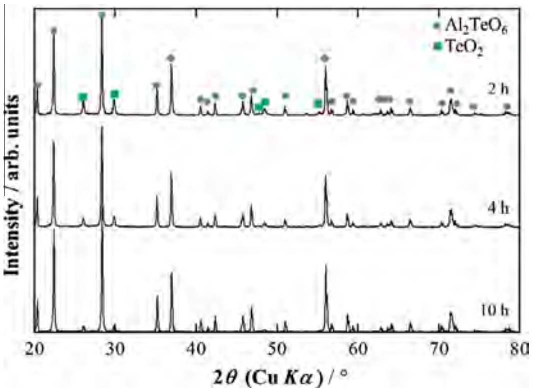

Fig. 3. XRPD patterns of the Al_2TeO_6 sintered for 2–10 h with additive 40 wt% TeO_2 at 900 °C.

true density of TeO_2 was higher than that of Al_2TeO_6. This is a main reason for the improvement of the apparent density by adding the TeO_2. However, the apparent density of the sample with the additive 50 wt% TeO_2 sintered at 900 °C for 2 h was lower than those of samples with additive 35–45 wt% TeO_2, which suggests that a greater amount of the TeO_2 evaporation makes the density lower for the 50 wt% case. The apparent densities for the 30 wt% increased initially and then decreased with the sintering time, although the 40 and the 45 wt% cases are apparently almost constant with the sintering duration time. As discussed below, this behavior might be related to both grain rearrangement and TeO_2 evaporation during sintering.

SEM images of the samples obtained by sintering the calcined Al_2TeO_6 with different amount of TeO_2 are presented in Fig. 6, where the samples were prepared by sintering the calcined Al_2TeO_6 with (a) additive 30-, (b) 40- and (c) 50-wt% TeO_2 at 900 °C for 2 h. The sample in the 30 wt% case has many pores, suggesting that the sintering is not well proceeded. With adding further TeO_2, the morphologies were obviously modified. Some grains are rearranged and aggregated for the cases of the 40 wt% and the 50 wt%. No remarkable difference in morphologies was observed between the 40 wt% case and the 50 wt% case. The rearrangement of grains is one of characteristics of liquid phase sintering [20]. The results mean that the TeO_2 takes a role of a liquid phase for the sintering, because TeO_2 is already melt at 900 °C, as described before. In the case of the additive 30 wt%, the amount of the liquid TeO_2 is not enough to wet and rearrange the Al_2TeO_6 grains.

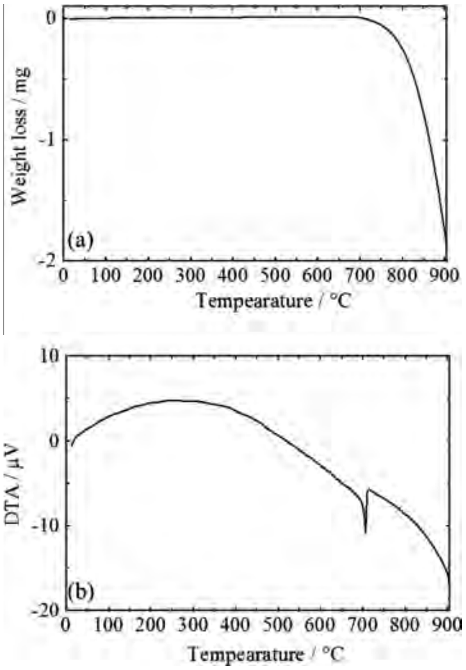

Fig. 4. Temperature dependence of the TG/DTA measurement of the Al_2TeO_6 with additive 40 wt% TeO_2.

SEM images of the samples by sintering the calcined Al_2TeO_6 with additive 40 wt% TeO_2 at 900 °C for (a) 2 h, (b) 3 h and (c) 5 h are portrayed in Fig. 7. With sintering duration time, the further rearrangement process seems to be proceeded. The rearrangement improves densification in this system. Thus the additional TeO_2 serves an important role in the densification of Al_2TeO_6–TeO_2 composite ceramics. However, pores were still observed in Fig. 7(c). As mentioned above, a part of TeO_2 is evaporated during heating at 900 °C. The evaporation makes further densification difficult. In Section 3.4, we will discuss a strategy to overcome this problem.

3.3. Microwave dielectric properties of the prepared Al_2TeO_6–TeO_2 samples

The relation between the dielectric constant ε_r and the preparation conditions ((1) additive TeO_2 amount and (2) sintering duration time) is presented in Fig. 8. The ε_r was investigated from the TE_{011} resonance mode observed at 13–14.5 GHz, depending on the preparation conditions. When the additive TeO_2 amount was 30 wt% and the sintering duration time was 2 h, ε_r was lower than those of the other prepared samples. Fig. 6(a) shows that the low ε_r resulted from poor densification of the sample. With an increasing amount of additional TeO_2 from 35 to 45 wt%, the ε_r increased slightly if one specifically examines the sintering duration time of 2 h. This fact suggests that the ε_r of the TeO_2 at about 13 GHz is higher than that of the Al_2TeO_6. Reportedly, that ε_r of TeO_2 is 19.3 at 4 GHz [3], which is higher than those of the Al_2TeO_6–TeO_2 composites in this study. As shown in the case of Al_2TeO_6, with additive TeO_2 of 35 or 40 wt%, the ε_r seems to decrease slightly with increasing sintering duration time from 2 h to 10 h. The TeO_2 evaporation is expected to contribute to the ε_r decrease.

The relation between quality factor ($Q \cdot f$) and the preparation conditions is presented in Fig. 9. The $Q \cdot f$ of the sample prepared with the condition: additive TeO_2 amount of 30 wt%, the sintering duration time of 2 h is the lowest among the prepared samples, resulting from the poor densification. If we specifically examined

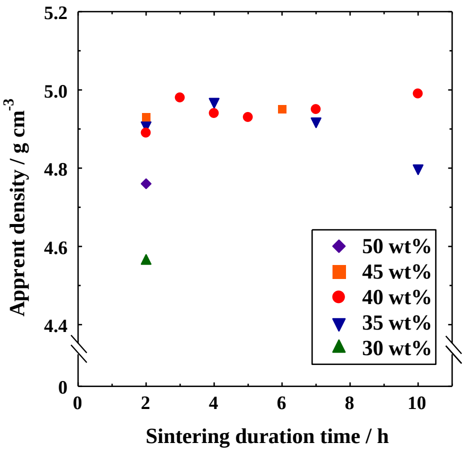

Fig. 5. Apparent densities of the prepared samples, where the calcined Al_2TeO_6 powders were sintered with the additive TeO_2 of 30–50 wt% at 900 °C for 2–10 h.

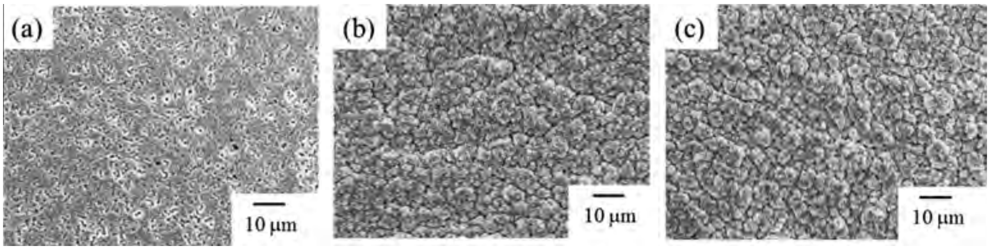

Fig. 6. SEM images of samples prepared by sintering the calcined Al_2TeO_6 with (a) additive 30 wt% TeO_2 at 900 °C for 2 h, (b) additive 40 wt% TeO_2 at 900 °C for 2 h, and (c) additive 50 wt% TeO_2 at 900 °C for 2 h.

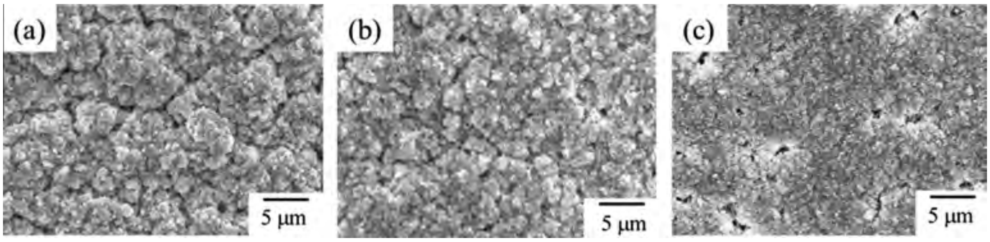

Fig. 7. SEM images of samples prepared by sintering the calcined Al_2TeO_6 with additive 40 wt% TeO_2 at 900 °C for (a) 2 h, (b) 3 h and (c) 5 h.

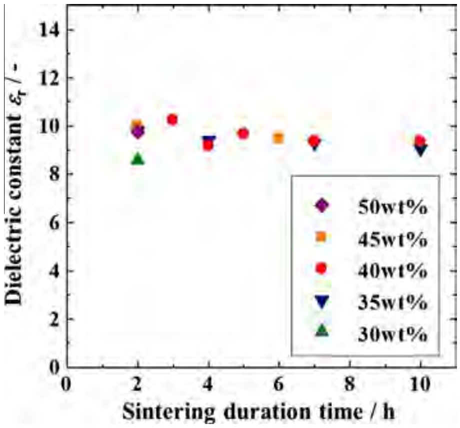

Fig. 8. Relation between dielectric constant ε_r and the preparation conditions ((1) additive TeO_2 amount and (2) sintering duration time), where the samples were sintered at 900 °C.

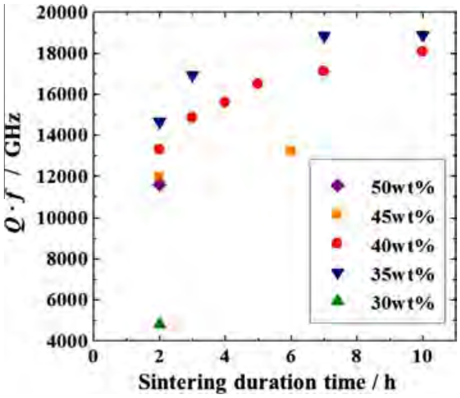

Fig. 9. Relation between quality factor and the preparation conditions ((1) additive TeO_2 amount and (2) sintering duration time), where the samples were sintered at 900 °C.

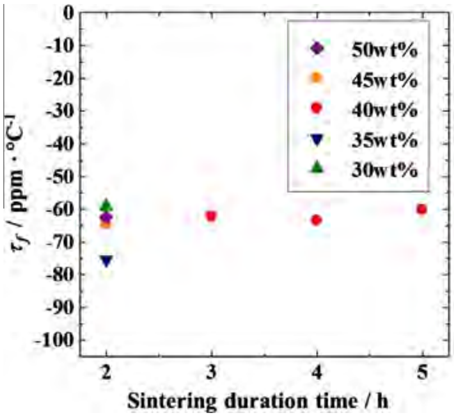

Fig. 10. Relation between τ_f and the preparation conditions ((1) additive TeO_2 amount and (2) sintering duration time), where the samples were sintered at 900 °C.

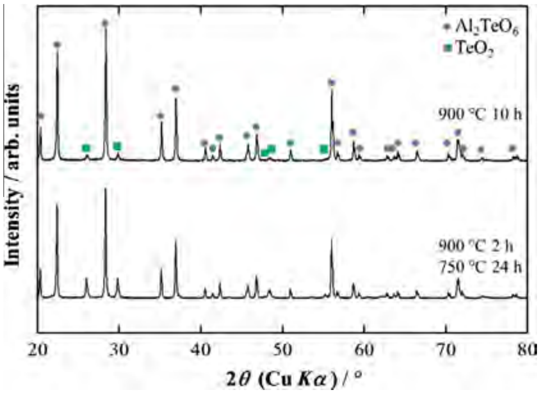

Fig. 11. XRPD pattern of the sample (Al_2TeO_6 with 40 wt% additive TeO_2) annealed at 750 °C for 24 h after sintering at 900 °C for 2 h. XRPD patterns of the sample sintered at 900 °C for 10 h (non-annealed sample) is also shown.

Table 1
Dielectric properties and the apparent density of the 40 wt% TeO_2–Al_2TeO_6 without and with annealing at 750 °C after sintering at 900 °C. The f_0 is the resonant frequency of the TE_{011} mode.

Sintering temp. (°C)	Sintering time (h)	Annealing temp. (°C)	Annealing time (h)	f_0 (GHz)	$Q \cdot f$ (GHz)	ε_r	Density (g/cm^3)
900	2	–	–	13.1	13,289	9.9	4.89
900	10	–	–	13.9	18,062	9.4	4.99
900	2	750	24	13.3	24,325	10.2	5.08

the preparation conditions: the additive TeO_2 amount of 35 and 40 wt%, $Q \cdot f$ for the 35 wt% case was higher than that for the 40 wt%. Although the apparent density was almost identical, which suggests that the $Q \cdot f$ of $AlTe_2O_6$ is higher than that of TeO_2. With increasing sintering duration time, $Q \cdot f$ increased gradually, as shown in the 35 or 40 wt% case. One reason for the increase is TeO_2 evaporation. Another reason is densification during maintenance of the temperature at 900 °C. Based on the results of the apparent densities, the ε_r, and the $Q \cdot f$, we presume that both densification and TeO_2 evaporation proceeded during heating at 900 °C.

The relation between τ_f and the preparation conditions is presented in Fig. 10. The τ_f values are in the range of −60 to −75 ppm/°C. No remarkable differences were investigated in the sample preparation conditions: (1) additives TeO_2 amount and (2) sintering duration time.

3.4. Effects of annealing at 750 °C on densification and microwave dielectric properties

As described above, sintering at 900 °C for a long time contributed to densification. However, longer duration times for sintering caused TeO_2 evaporation. To overcome this problem, we investigated an annealing effect after sintering at 900 °C for 2 h. The XRPD pattern of the sample (Al_2TeO_6 with additive 40 wt% TeO_2) annealed at 750 °C for 24 h after sintering at 900 °C for 2 h is presented in Fig. 11. For comparison, XRPD of the sample sintered at 900 °C for 10 h is also shown. After annealing, no other secondary phase or no peak shift was observed, suggesting that influence of annealing on reduction or oxidation is small in this composite system. The TeO_2 reflection intensities of the annealed samples were higher than that of the sample sintered at 900 °C for 10 h. The result supposes that the annealing is effective to make TeO_2 evaporation slower. Apparent densities and dielectric constants of the annealed samples were compared to those of the non-annealing samples. The results are tabulated in Table 1. The $Q \cdot f$ in the case of the annealing at for 24 h was higher than in the non-annealing sample sintered at 900 °C for 10 h. Apparent densities and dielectric constants for the annealing sample were higher than those of the non-annealing samples. These facts suggest that sintering proceeded well during annealing because of the slower TeO_2 evaporation at 750 °C than at 900 °C. The sintering proceeding during slower TeO_2 evaporation contributes to higher quality factors.

4. Conclusions

We investigated a low-temperature sintering condition for Al_2TeO_6–TeO_2 ceramics and their microwave dielectric properties. Heating temperature of 620 °C is an appropriate condition to prepare the Al_2TeO_6 calcined powders. The calcined Al_2TeO_6 powders were sintered with 30–50 wt% additive TeO_2 at 900 °C for 2–10 h. XRPD patterns showed that the sintered samples were Al_2TeO_6–TeO_2 composite with no other phase. The apparent density was improved by adding the TeO_2 of up to 45 wt%. Particularly, the apparent density of Al_2TeO_6 with an additive 40 or 45 wt% TeO_2 was higher among the prepared samples sintered at 900 °C for 2 h. The dielectric constant ε_r of the Al_2TeO_6–TeO_2 composite increased slightly with TeO_2 content from 35 to 45 wt%, suggesting that the ε_r of TeO_2 is higher than that of Al_2TeO_6. The $Q \cdot f$ of the Al_2TeO_6–TeO_2 composite decreased with TeO_2 content from 30 to 45 wt%, suggesting that the $Q \cdot f$ of TeO_2 is lower than that of Al_2TeO_6. During sintering at 900 °C, the ε_r of the Al_2TeO_6–TeO_2 decreased slightly, whereas the $Q \cdot f$ increased gradually. As shown in the SEM image, the longer sintering time makes pores fewer and denser the Al_2TeO_6–TeO_2 ceramics. These facts mean that the sintering at 900 °C for a long time contributed to densification, however, simultaneously causing the TeO_2 evaporation. To prevent the TeO_2 evaporation, we investigated effects of annealing at 750 °C for 24 h after sintering at 900 °C. Apparent densities or ε_r for the annealing samples were higher than those of the non-annealing samples. The $Q \cdot f$ was improved by annealing, suggesting that sintering proceeded well during annealing because of slower TeO_2 evaporation at 750 °C.

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