Journal of the Korean Ceramic Society
Vol. 40, No. 4, pp. 350~353, 2003.

Millimeter-wave Dielectric Ceramics of Alumina and Forsterite with High Quality Factor and Low Dielectric Constant

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(Received March 19, 2003; Accepted April 7, 2003)

ABSTRACT

Millimeter-wave dielectric ceramics have been used like applications for ultrahigh speed wireless LAN because it reduces the resources of electromagnetic wave, and Intelligent Transport System (ITS) because of straight propagation wave. For millimeter-wave, the dielectric ceramics with high quality factor (Q·f), low dielectric constant(ε_r), and nearly zero temperature coefficient of resonant frequency (τ_f) are needed. No microwave dielectric ceramics with these three properties exist except $Ba(Mg_{1/3}Ta_{2/3})O_3$ (BMT), which has a little high ε_r. In this paper, alumina (Al_2O_3) and forsterite (Mg_2SiO_4), candidates for millimeter-wave applications, were studied with an objective to get high Q·f and nearly zero τ_f. For alumina ceramics, Q·f more than 680,000 GHz was obtained but it was difficult to obtain nearly zero τ_f. On the other hand, for forsterite ceramics, Q·f was achieved from 10,000 GHz of commercial forsterite to 240,000 GHz of highly purified MgO and SiO_2 raw materials, and τ_f was reduced a few by adding TiO_2 with high positive τ_f.

Key words : Millimeter-wave dielectric materials, Microwave materials, Alumina, Forsterite, Quality factor

1. Introduction

Recently, microwave telecommunication has been developed for wide applications, such as mobile phone, wireless LAN and Intelligent Transport System(ITS). Microwave dielectric materials are expected to be developed for variety of application including miniaturization for mobile phone, transmitter and receiver with high performance for base station, and ultrahigh speed wireless LAN and ITS for millimeter wave range. The development trend of microwave dielectric materials is schematically shown in Fig. 1. In this figure, Quality factors (Q·f) measured in variety of microwave dielectric materials are shown as a function of their dielectric constants (ε_r). Both Q·f and ε_r are two of three key factors describing dielectric property. Q is inverse of the dielectric loss (tan δ) and large ε_r shortens wavelength, as followed by the relation: $\lambda=\lambda_0/\varepsilon_r^{1/2}$. The temperature coefficient of resonant frequency (τ_f) is the other key factor describing microwave dielectric property. τ_f is expected to be close to zero.

In particular, millimeter-wave dielectric ceramics have been recently attracted much attention. They are planned to be applied in ultrahigh speed wireless LAN, because they can reduce the resource of electromagnetic wave. Millimeter-wave dielectric ceramics are also planned to be used in the ITS including car anti-collision system *etc.*, by using excellent wave property of straight propagation. Further, there are much needs to reduce cross-coupling between microstrip lines on circuit boards, and to minimize signal delay times. The signal propagation speed is inversely proportional to ε_r of IC dielectric substrate, so that performance is enhanced as ε_r decreases.[1)] For these applications, dielectric ceramics with high Q·f and low ε_r are strongly needed, and nearly zero τ_f is also needed for more high quality applications. None of the presently available microwave dielectric ceramics can satisfy all of these three factors

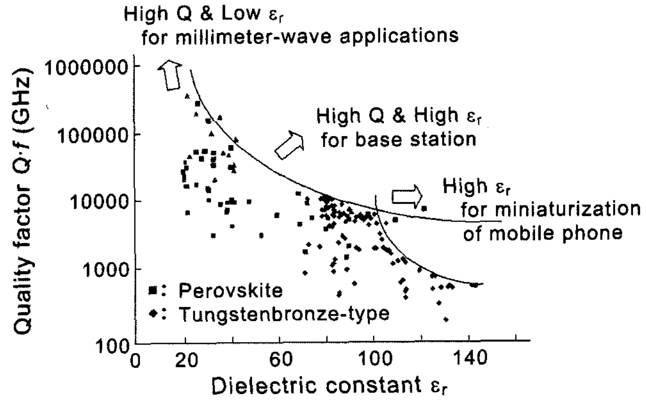

Fig. 1. Directons of development of micowave dielectrics desighned on a figure: Q·f as a function of ε_r.

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except for $Ba(Mg_{1/3}Ta_{2/3})O_3$ (BMT) which shows a little higher ε_r than expected for millimeter-wave application.

Alumina (Al_2O_3) and forsterite (Mg_2SiO_4) ceramics, which show much lower ε_r than BMT, are also candidates for millimeter-wave applications. Especially, alumina ceramics has ultrahigh Q·f of 360,000 GHz and raw ε_r of 9.8.[2,3] Moreover, alumina has been used as insulator such as substrate and package materials because of its mechanical hardness.[4] On the other hand, forsterite has lower ε_r than alumina. Forsterite ceramics have been used as electronic parts such as resistor core, electron tube stem, base parts supporting dielectric resonator *etc.*, because of the low ε_r and high insulation resistance even in the microwave frequency range. Therefore, it is also expected as a candidate for the application to IC dielectric substrate.

In this study, alumina and forsterite ceramics were synthesized by using high-purity and fine-grain-size law powders to obtain excellent materials with high Q·f and nearly zero τ_f. On the other hand new designed forsterite ceramics were synthesized by using highly purified MgO and SiO_2 raw materials.[5] Moreover, reduction in τ_f was attempted by adding a few amounts of TiO_2 with high positive τ_f into forsterite ceramics.[6]

2. Experimental Procedures

High purity (99.99%) and fine grain (170 mm) powder, Taimicron TM-DAR (Taimei Chemical Co., Ltd.) was used for synthesis of alumina ceramics. Pellets with diameter of 14 mm were formed by uni-axial pressure of 98 MPa after wet ball-milling of the powder and sintered at temperature from 1350 to 1600°C for 5 h in air.

To prepare forsterite ceramics, high purity (99.99%) MgO powder manufactured by gas-phase oxidation process of magnesium was used. The powder showed single crystal grains with fine particle size distribution in the range of 0.08 to 0.1 mm and large specific surface area of 2,600 m^2kg^{-1}. High purity SiO_2 powder was also used for synthesis of forsterite ceramics. The powder has 99.8% purity and particle size of 1.6 mm and specific surface area of 4,000 m^2kg^{-1}. These powders mixed in an urethane ball mill for 20 h in distilled water, then freeze-dried. The powders were calcined at 1200°C for 3 h. The calcined powders were again ball-milled for 24 h to produce forsterite powders with an average particle diameter of about 1 mm. The pellets with diameter of 15 mm were formed by CIP at 300 MPa. The pellets were sintered at temperature from 1300 to 1450°C for 2 h.

The crystalline phases of sintered pellets are investigated by X-ray powder diffraction. The relative density was measured by the Archimedes' method. The microstructure was observed by a Scanning Electron Microscope (SEM). The microwave dielectric properties (ε_r, Q·f, and τ_f) of alumina ceramics were estimated using a pair of parallel conducting Ag plates in the TE_{011} mode, using Hakki and Colemanís method,[7] and those of forsterite also at 23 GHz using JIS R 1627-1996, by network analyzer.[8]

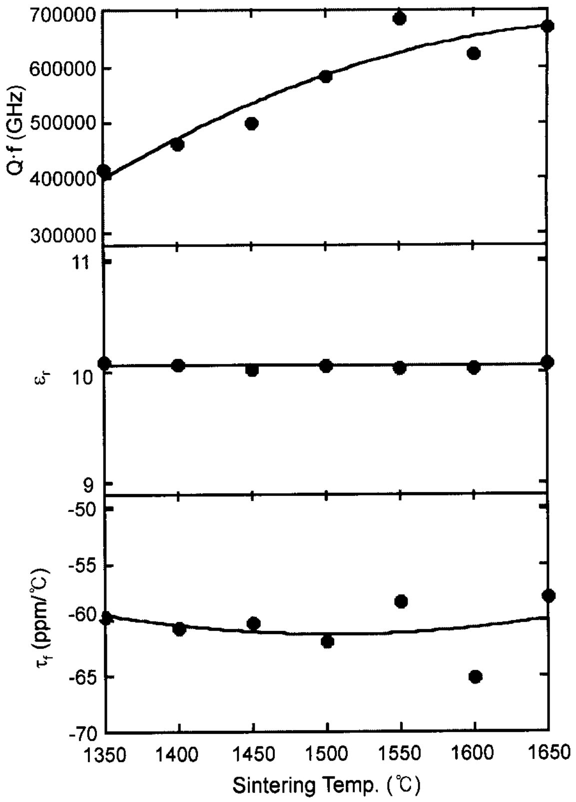

Fig. 2. Microwave dielectric properties of alumina ceramics obtained.

3. Results and Discussion

3.1. Alumina Ceramics

Microwave dielectric property of alumina ceramics as a function of sintering temperature is shown in Fig. 2. Q·f value becomes higher as the temperature increases. In the temperature range higher than 1500°C, the large Q·f value larger than 600,000 GHz is observed. The highest Q·f value of 680,000 GHz was obtained in the temperature range of 1550 to 1650°C and in the case of 5 h sintering. This value is the highest Q·f in the world as far as we know for single-phase alumina ceramics, although the Q·f value of 1,000,000 GHz was reported in alumina single crystal. High Q in the alumina ceramics is considered to result from high purity and high density. Relative density and SEM micrographs as a function of sintering temperature are shown in Figs. 3 and 4, respectively. High relative density more than 99.5% was obtained for each sample, but grain growth was observed in the sample sintered at 1600°C, compared with the sample sintered at 1350°C. On the other hand, ε_r of 10.05 and τ_f of −60 ppm/°C are not affected by different sintering temperature.

3.2. Forsterite Ceramics

Q·f values of forsterite ceramics as a function of sintering temperature are shown in Fig. 5. The highest Q·f of 240,000 GHz was obtained at sintering temperature of 1360°C. This

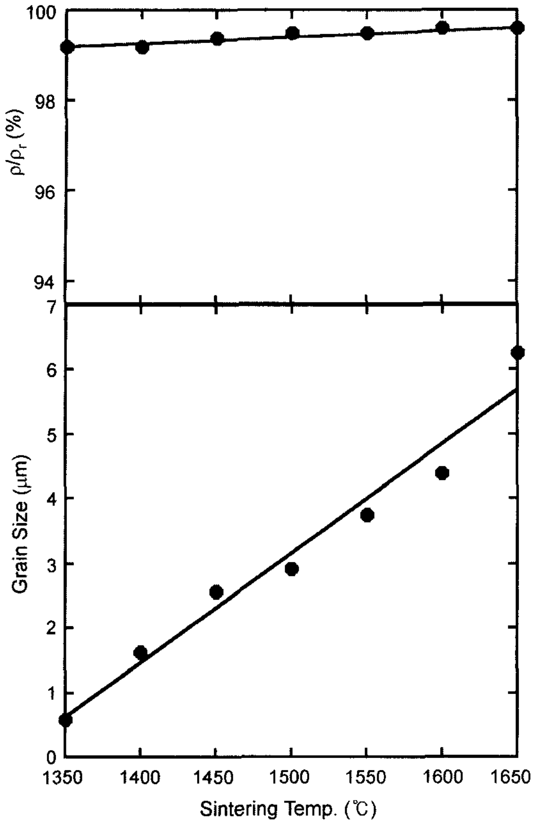

Fig. 3. Relative densities and grain sizes of alumina ceramics obtained.

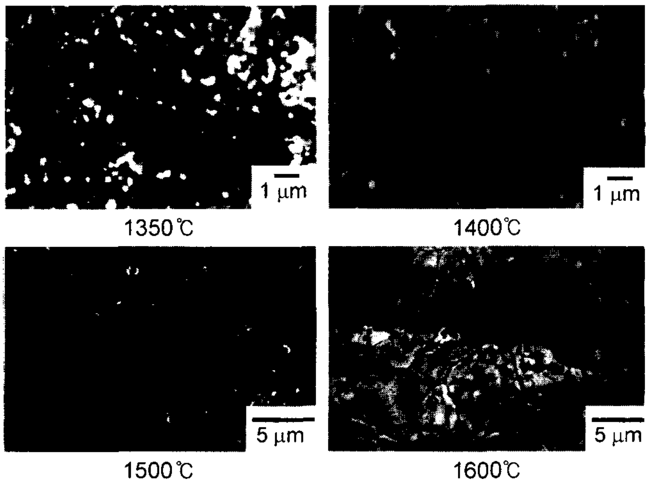

Fig. 4. SEM photographs of alumina ceramics obtained with different sintering temperature in which the grain sizes increase according to sintering temperature.

Q·f value is much higher than that of commercially available forsterite ceramics as shown in Fig. 6. The density of the sintered sample showed 96–98% of theoretical density. The sample was single crystalline phase of forsterite with no glassy phase at the grain boundaries, which is believed to reduce dielectric loss significantly into the low level measured in the single crystal. Its SEM micrograph is shown in Fig. 7. The grain growth improved the Q·f in fine forsterite ceramics. For comparison, a micrograph of commercially available forsterite is also shown in this figure. The grain boundary observed in the commercially available forsterite is thick and includes many impurities and glassy phases, which resulted in large dielectric loss. The dielectric constants are smaller than 6.90 which is expected to bring high speed electromagnetic-wave propagation, protecting delay of signal propagation.

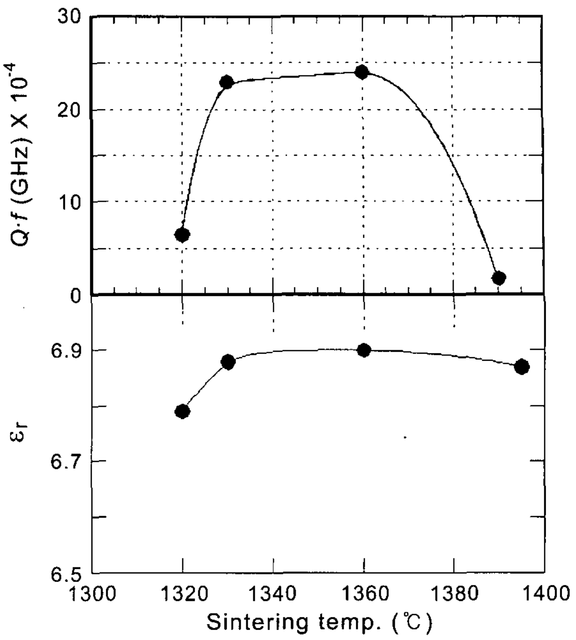

Fig. 5. Q·f and ε_r of forsterite as a function of sintering temperatures.

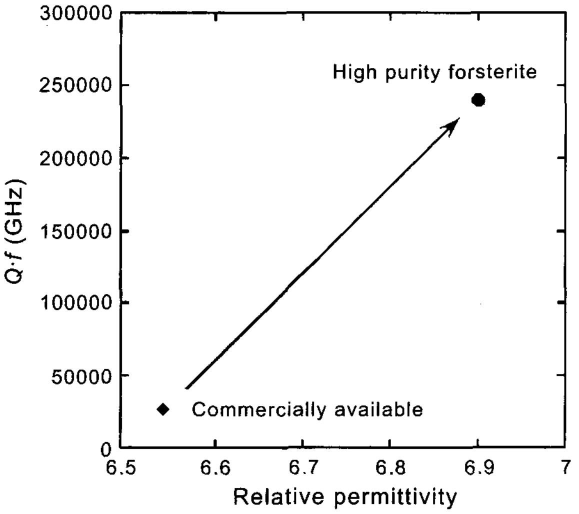

Fig. 6. Q·f is improved in high purity forsterite ceramics.

The temperature coefficient of resonant frequency τ_f of forsterite was estimated to be −67 ppm/°C which was calculated using following equation:

$$\tau_f = -(\alpha + \tau_\varepsilon/2) \qquad (1)$$

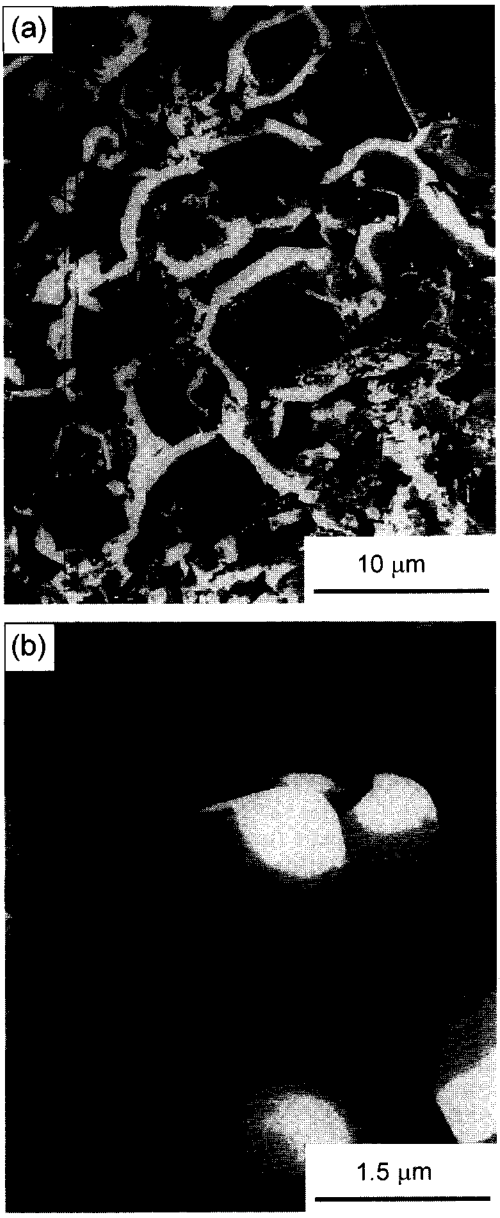

Fig. 7. SEM photographs of commercially available (a) and high purity forsterite ceramics (b).

where, τ_e is temperature coefficient of dielectric constant (ε_r) and α is thermal expansion coefficient. They were 116 ppm/°C and 9.4×10^{-6} in the range of 25~700°C, respectively. The τ_f value was attempted to be reduced close to zero by means of adding rutile (TiO_2) having positive τ_f of 450 ppm/°C. However, τ_f could be reduced only a little amount from −67 ppm/°C to −63 ppm/°C, even at a large amount (30 wt%) of TiO_2 addition. This result comes from phase equilibrium in the MgO-SiO_2-TiO_2 ternary system. The composition with 30 wt%TiO_2 locates in a Mg_2SiO_4-$MgTi_2O_5$-ternary subsystem, then TiO_2 disappeared. For zero τ_f resonator, the sintering processing should be considered.

4. Conclusions

Alumina and forsterite ceramics for candidates millimeter-wave dielectrics with high Q and low ε_r were presented in this paper. Q·f factors of alumina ceramics were improved to 680,000 GHz by using high purity and fine grain size alumina raw materials. Forsterite ceramics were also improved to 240,000 GHz by using MgO raw material. Dielectric constant of forsterite is 7.8 smaller than alumina ceramics. Low dielectric constant reduces delay of signal propagation. Moreover, we tried temperature coefficient of resonant frequency (τ_f) to near zero τ_f by means of adding TiO_2 with high positive τ_f. But, τ_f did not improved because of TiO_2 disappeared during reaction based on phase equilibrium. Ceramics with near zero τ_f should be created near future.

Acknowledgment

The authors thank Dr. Sundarakannan B and students of NIT for preparing this paper with figures. A part of this study are supported by government special budget consortium project planned by Japanese Ministri of Economy, Trade and Industry, and The 21st. COE project planed by Japanese Ministry of Education, Science and Culture in Japan.

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