Microwave Processing and Properties of Ceramics with Different Dielectric Loss

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Abstract

Microwave sintering behaviors of three kinds of ceramics with different dielectric loss [Al_2O_3, Ce–Y–ZrO_2 and lead-based relaxor ferroelectrics (PMZNT)] in 2·45 GHz microwave furnace were described. Measurement of sample densities showed an enhancement of the sintering processing for all materials studied. For PMZNT and Ce–Y–ZrO_2 with high dipolar loss or ionic conductive loss, the associated microstructure examined using scanning electron microscopy showed that microwave-sintered compacts produced much finer grain sizes at near theoretical density compared to conventional sintering. Resulting material properties, such as flexure strength and breakdown strength, were also increased due to developed microstructure in microwave processing. However, a comparable grain size and properties were observed for high pure Al_2O_3 with low dielectric loss in microwave and conventional methods. © 1999 Elsevier Science Limited. All rights reserved

Keywords: microwave processing, microstructure – final, Al_2O_3, ZrO_2, ferroelectric properties.

1 Introduction

Microwave processing of ceramics has gained much attention during last decade. Reasons for the growing interest in the use of microwave energy include rapid heating, enhanced densification rate, decreased sintering active energy and improved microstructure.[1–3] Microwave heating also has the potential for energy and cost savings when compared with conventional heating.[4,5]

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Electromagnetic waves interact with ceramic materials, leading to volumetric heating by dielectric loss. There are two main physical loss mechanisms: the flow of conductive current (in particular ionic conduction), and dipolar reorientation. Mathematically both of these losses may be included in an effective dielectric loss factor

$$\varepsilon_e'' = \varepsilon'' + \frac{\sigma}{\omega\varepsilon_0} \qquad (1)$$

where ε'' is the dielectric loss factor, representation of dipolar losses; σ is the conductivity; ε_0 so is the permittivity of free space; ω is the frequency; $\frac{\sigma}{\omega\varepsilon_0}$ presents conductive losses. The power deposited in the ceramics is given by

$$P = \omega\varepsilon_0\varepsilon_e''/E/^2 = \omega\varepsilon_0\left(\varepsilon'' + \frac{\sigma}{\omega\varepsilon_0}\right)/E/^2 \qquad (2)$$

where E is the electric field.

There are differences in microwave adsorption levels for various ceramics due to their different crystal structure and loss mechanisms. Also the degree of interaction between microwave and ceramics changes with temperature.[6] Most of structure ceramics behavior as conductive loss mechanism in microwave field, such as Al_2O_3, ZrO_2 and Si_3N_4. They have a low absorption ability at lower temperature and a increased absorption at higher temperature. Earlier work by Meek *et al.*[7] showed that microwave sintering of ZrO_2–Y_2O_3 and pure Al_2O_3 reached 93·3 and 91·7% theoretical density respectively. In order to accelerate initial heating at low temperature and get more uniform heating, Janney *et al.*[8] used multimode cavity with 'picket fence' arrangement and successfully sintered ZrO_2–8 mol% Y_2O_3 compacts to high density (> 99% theoretical). A similar result was reported for pure alumina.[9] For microwave sintering of electronic ceramics, many of them, especially ferroelectric and piazeloelctric materials, can couple microwave energy by both conductive losses and dipolar losses. This make them

heated in microwave field more effective. Harrison[10] showed that PZT and PLZT can be densified in air with fast heating and short hold time of 5 min. Varadan et al.[11] rapidly sintered $SrTiO_3$ to a density of 98% by microwave heating to 1300°C at 3°C s^{-1} whereas conventional heating (0·08°C/s) resulted in a sintered density of 80%.

As mentioned above, microwave sintering behaviors of ceramics strongly rely on their loss mechanism and adsorption ability. In the present work, comparable study of microwave processing for three kinds of ceramic materials with different adsorption, Al_2O_3, Ce–Y–ZrO_2 and lead-based relaxor ferroelectrics with compositions of $xPb(Mg_{1/3}Nb_{2/3})O_3$-$yPb(Zn_{1/3}Nb_{2/3})O_3$-$zPbTiO_3$ (PMZNT), was carried out. The effect of microwave heating on final microstructure and grain size of these materials was discussed. Also their properties after sintering were investigated.

2 Experimental Procedure

2.1 Sample preparation
A high purity (99·97%) Al_2O_3 powder (Ceralox APA, Ceralox Corporation, Touscon USA) with an average particle size of 0·4 μm and BET of 10 m^2/g was used. The 5 wt% Ce–3 wt% Y–ZrO_2 powder was prepared by the aqueous chemical coprecipitation method, and the particle size ranged from 0·05 to 0·1 μm. X-ray diffraction (XRD) analysis showed that monoclinic-phase was found for the start Ce–Y–ZrO_2 powder. Powder above was wet-ball milled with 3 mol% PVA. After dried and sieved, the powder containing binder was first uniaxially pressed at 100 MPa and then isostatically pressed at about 250 MPa into the samples with 5 × 6 × 45 mm^3 or 6 × 8 × 45 mm^3. The binder of the green compacts was then burn out in a muffle furnace, and resulting compacts had green density of 57 and 52% for Al_2O_3 and ZrO_2. Lead-based relaxor ferroelectrics powder with compositions of $xPb(Mg_{1/3}Nb_{2/3})O_3$-$yPb(Zn_{1/3}Nb_{2/3})O_3$-$zPbTiO_3$ (PMZNT) was prepared as described as previous work.[12] The powder was consolidated by uniaxial/isostatically pressing into two size samples: (1) disks with 15 × 1 mm thickness for dielectric breakdown strength, and (2) bars with 6 × 5 × 40 mm^3 for flexural strength. The green compacts had a density of approximately 60%.

2.2 Microwave sintering
The green samples were sintered in air using 2·45 GHz microwave energy. The microwave sintering system is consisted of 0·78 × 10^{-2} m^3 multimode cavity and a continually adjustable power supply of 0·5–5 kW, which has been illustrated indetail elsewhere.[13,14] SiC rods (picket fence) susceptors was used to initially hybrid heat low-loss samples at relative lower temperature. The arrangement of samples and 'picket fence' susceptors/thermal insulation was shown in Fig. 1 Temperature was measured in the range of 550–2000°C using far-inferred fiber optic pyrometer as reported by Katz.[15] The temperature was detected by the pyrometer and transformed to temperature signal. For comparison, the same samples were also sintered by a conventional method with a fast heating rate of 200°C h^{-1}.

2.3 Property measurement
Density of sintered-specimens were measured by the Archimede's method. Microstructure of the samples was observed by SEM (computerized scanning electron microscope, Model 950, OPTON, Germany). The average grain size of the polished and thermally etched surface was determined by linear intercept method.[15] X-ray diffraction (XRD) was used to analyze the phase composition of the polished ZrO_2. The fraction of tetragonal-phase zirconia was determined.[15] Flexural strength was measured by a three-point bending test, and fracture toughness was measured by a single-edge notched beam test with a notch width of 0·22 mm and a span of 24 mm. The dielectric breakdown strength of PMZNT was measured with D.C power supply with high stability where samples with thickness of 0·30–0·35 mm were used.

3 Results and Discussion

3.1 Microwave heating behaviors
A constant power of about 0·5 kW was applied for an initial few minutes, and then was gradually increased to the sintering temperature. Typical heating profiles of the three materials were shown in Fig. 2 Clearly, more rapid heating rate with
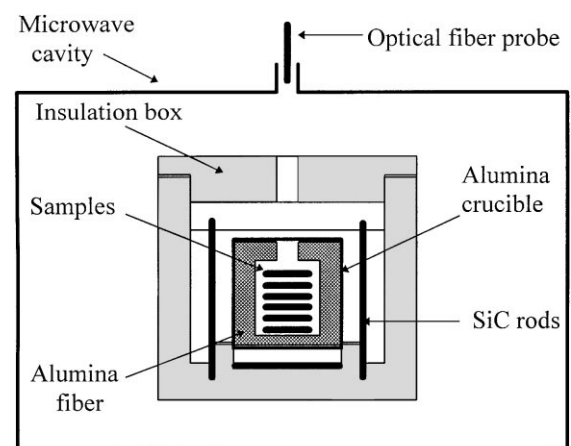

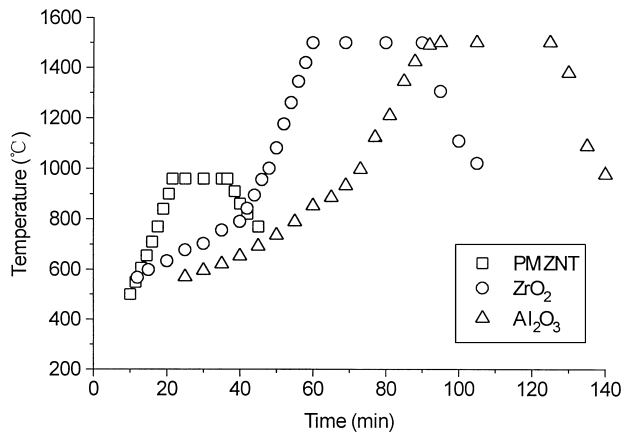**Fig. 1.** Microwave hybrid heating structure.

Fig. 2. Typical microwave heating profiles.

Fig. 3. Ceramic conductivity changes with temperature.

about 50°C min^{-1} was reached for the PMZNT ferroelectrics. This is due to that the materials with Perovskite structure exhibits a strong dielectric loss, in particular dipolar losses. Microwave heating of Al_2O_3 and ZrO_2, however, mainly depend on their conductive loss.[16] Also they have lower conductive losses compared to other semiconductive ceramics and ferroelectrics, especially at low temperature. Therefore, a slow heating rate was observed for Al_2O_3 and ZrO_2 at lower temperature. During these heating periods, heating to the samples was mainly from SiC rod susceptor because they exhibit more strong absorption to microwave field compared with samples. After reaching critical temperature (Tc), the samples coupled with the electromagnetic field more effectively and higher heating rate was obtained due to rapid increase of ionic conduction and absorption ability,[17] and sufficient depth of microwave penetration to samples can be maintained. However, the depth of microwave penetration of SiC rods was greatly decreased from 4 cm (at 22°C) to 0·4 cm at 695°C,[9] the heating of susceptors on samples is less compared to that at relative lower temperature. Also it was measured that samples in picket fence showed much higher temperature than SiC rods during sintering stages. These results revealed that uniform microwave sintering of the samples mainly was produced by their interaction with the electromagnetic field.

In addition, Fig. 2 also shows there is difference for the critical temperature and time to reach the critical temperature for Al_2O_3 and ZrO_2 samples. Since Al_2O_3 has less ionic conduction and dielectric loss (shown in Fig. 3) it tends to have a higher critical temperature and needs more time to reach final temperature compared with ZrO_2.[17]

Many researches[18,19] have shown thermal runaway in microwave-heated material often occurred at above critical temperature. It generally leads to thermal stress and cracking of ceramics, especially for low thermal conductive ceramics such as Al_2O_3.
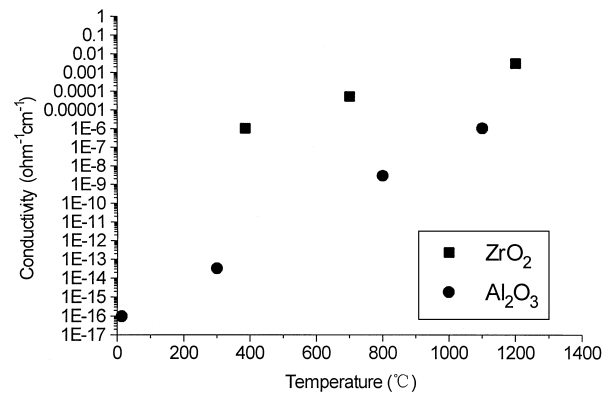
Therefore, the heating rate should be carefully controlled by varying the input microwave power. ZrO_2 with high thermal conductivity, however, tends to sinter more rapidly and more effectively in a microwave field than Al_2O_3. In the cases of the three materials, thermal runaway and cracking were successfully avoided by controlling the heating rate and input power as well as the use of 'picket fence' susceptors/thermal insulation.

3.2 Densification and microstructure

Density variation with temperature for the three kinds of materials in microwave and conventional sintering was shown in Figs 4–6, respectively. It can be found that the microwave-sintered samples exhibit enhanced densification compared to their conventional-sintered samples at low sintering temperature for these materials. Microwave-sintered Ce–Y–ZrO_2 (shown in Fig. 5), for example, reached a 94% theoretical density at 1300°C with a hold of 30 min, whereas a conventionally-sintered sample reached the same density at 1400°C with a held of 2 h. A temperature difference of 100°C was observed between microwave and conventional processing for reaching the density. Similar results were observed for Al_2O_3 (in Fig. 4). In the case of PMZNT (shown in Fig. 6), microwave fast sintered samples (15 min holding time) reached much higher density than that of conventional sintering with long holding time of 4 h at low temperature.
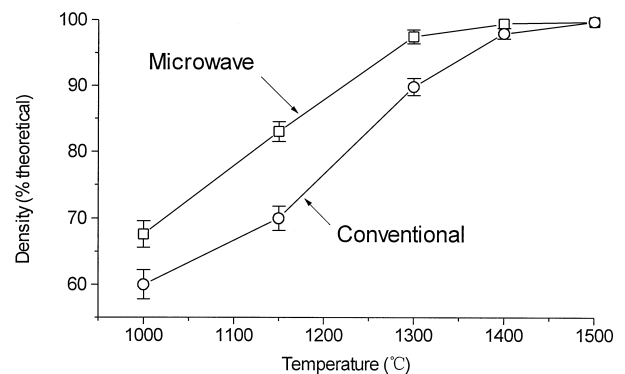

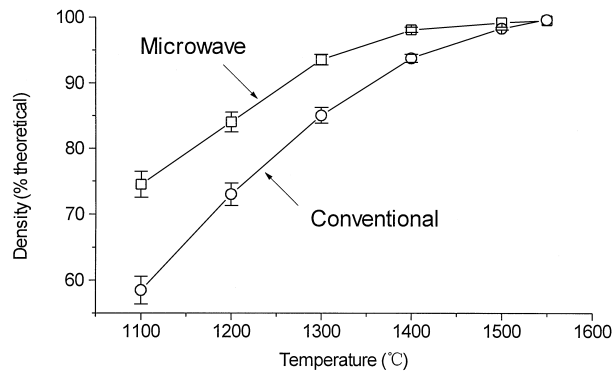**Fig. 4.** Density variation with sintering temperature for Al_2O_3.

Fig. 5. Density variation with sintering temperature for Ce–Y–ZrO$_2$.
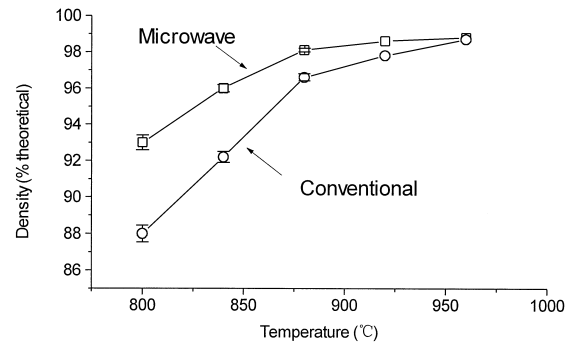

Fig. 6. Density variation with sintering temperature for PMZNT.

Clearly considerable dipolar losses and conductive losses of the materials should be responsible for rapid desiccation rate. It is noted, however, the density differences between microwaved and conventionally sintered samples tend to decrease with increased temperature for all these materials. Finally, the almost same density was obtained at final sintering temperature. This implies that microwave heating can accelerate densification, save sintering time or reduce temperature, but not shows higher density was achieved than conventional method at final sintering temperature. Similar results were observed by other research.[20] Previous work[21] on Y–TZP with agglomerate also showed microwave and conventional sintered samples had the same density of 96% theoretical at final temperature.

The mechanism of microwave fast sintering is not clear now, but several explanations were proposed. Janney and Kimrey[18] thought microwave sintering can reduce apparent energy for Al$_2$O$_3$ and ZrO$_2$. They report activation energies of 160 and 570 kJ mol^{-1} for microwave and conventional sintering, and 410 and 710 kJ mol^{-1} for ^{18}O tracer diffusion in alumina, using microwave and conventional heating, respectively. Fathi *et al.*[22] attributed the enhanced diffusion to increased vibrational frequency of the ions caused by the electric field of the microwave radiation. Booske *et al.*[23] proposed that the effect of the microwaves would be to excite a non-thermal phonon distribution in the polycrystalline lattice. This would translate into a non-thermal energy distribution, thereby enhancing the mobility of crystal lattice ions. Recent calculations by Binner[24] showed that the faster diffusion rates might be explained an increase in the Arrhenius pre-exponential factor A, with no change in the activation energy.

Scanning electron microscopy was used to examine the microstructures of both microwave and conventional samples. Figure 7 shows micrographs of fracture surface of these samples with high densification of 99% theoretical. For the comparison of the three materials, clearly, microwave sintering of relaxor ferroelectrics produces a product with a much smaller and more uniform grain structure [shown in Fig. 7(a)]. The average grain size of the microwave-sintered sample was found to be 3·2 μm whereas 6·8 μm was observed in the conventionally-sintered sample [in Fig. 7 (d)]. The big difference in grain size implies that microwave sintering has the potential of suppressed grain growth due to a fast heating rate and a rapid enhanced diffusion. In the case of ZrO$_2$, a similar result was obtained. The grain sizes of 0·65 and 1·2 μm were observed in microwave and conventionally sintered samples [shown in Fig. 7(b) and (e)], respectively, where a ratio of grain size was about 0·6. This ratio is in agreement with that of Y–TZP reported by Janney *et al.*[20] Microwave-sintered Al$_2$O$_3$ sample [in Fig. 7(c)], however, has almost same grain size ($\sim$2·5 μm) compared with conventional sintered sample [in Fig. 7 (f)] at density of more than 99% theoretical. Table 1 shows variation of grain size versus temperature for Al$_2$O$_3$ and ZrO$_2$. It can be seen rapid grain growth also was accompanied with the enhanced densification in microwave sintering of Al$_2$O$_3$. But the grain growth of ZrO$_2$ in microwave processing has less increase than that in conventional heating. The above results suggest that the reduction of grain size in microwave-sintered materials is influenced by their dielectric loss. The grain sizes of relaxor ferroelectrics and zirconia were significantly reduced in microwave processing due to their high dipolar losses or conductive losses. This feature shows there are different densification or diffusion mechanism for the materials with various loss in microwave processing.

3.3 Improved properties

Properties of the three materials sintered in microwave and conventional methods were shown in Table 2. In the cases of Ce–Y–ZrO$_2$ and relaxor ferroelectrics (PMZNT), improved properties were obtained for microwave sintering. Microwave
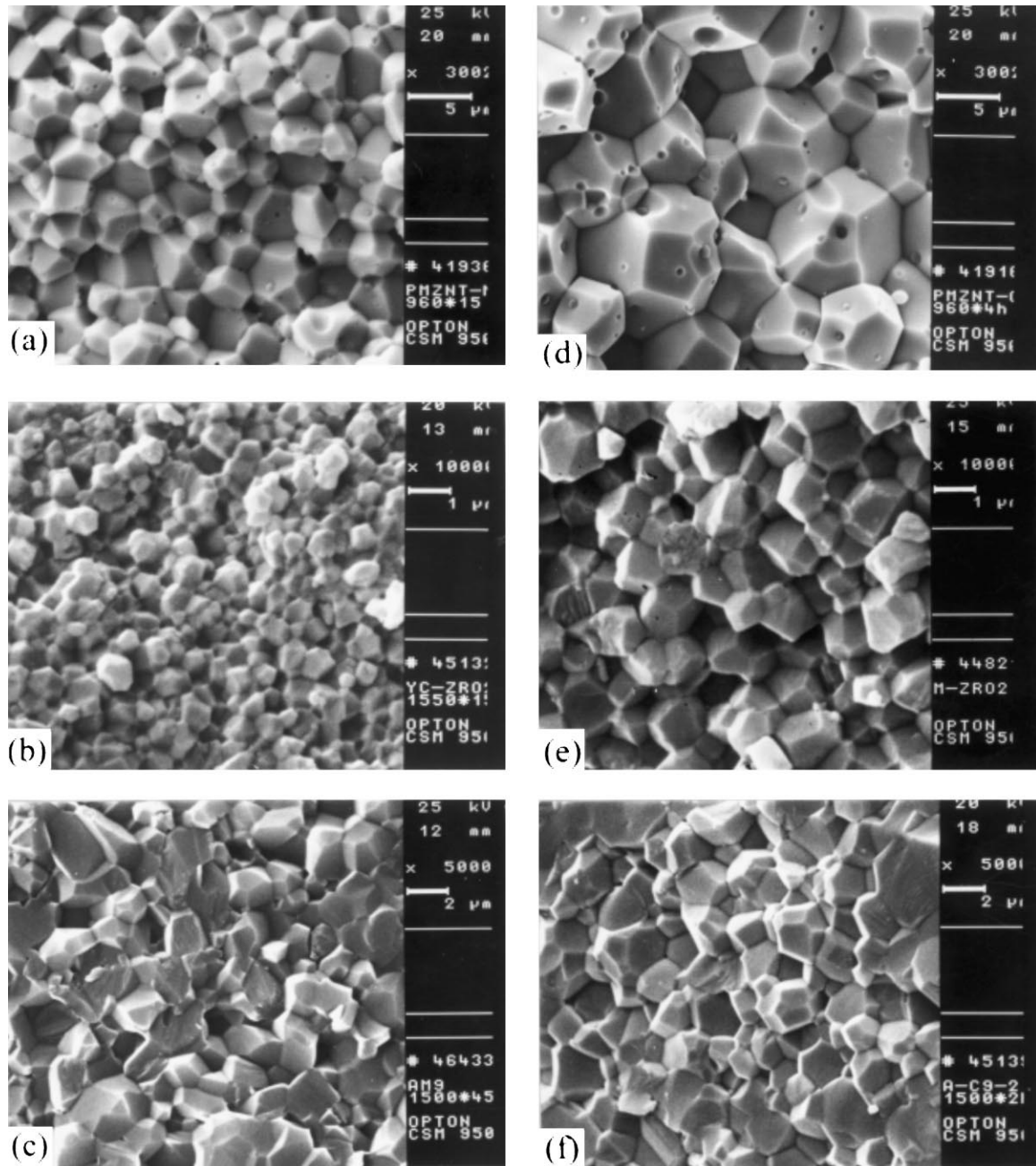
Fig. 7. SEM micrographs of samples sintered in microwave furnace: (a) PMZNT; (b) CE–Y–ZrO_2; (c) Al_2O_3, and in conventional furnace; (d) PMZNT; (e) Ce–Y–ZrO_2; (f) Al_2O_3.

sintering of the relaxor ferroelectrics, for example, can reach higher flexure strength ($\sim$90 MPa) than conventional sintering ($\sim$65 MPa). And about 40% strength increase was achieved for the microwave method. Similarly, high breakdown strength of 105 kV mm^{-1} was reached in microwave-sintered sample whereas 62 kV mm^{-1} in conventional. This means defect size in the material was reduced due to a more developed uniform and finer grain microstructure in microwave processing. For the material of C–Y–ZrO_2, flexural strength and fracture toughness also were increased from 1160 MPa and 12·6 MPa m$^{1/2}$ in conventional heating to 1200 MPa and 13·7 MPa m$^{1/2}$ in microwave heating, respectively. In addition to finer grain size and

microstructure, more tetragonal phase zirconia was also produced in microwave sintering than in conventional sintering. Ninety-six per cent tetragonal zirconia was obtained in the polished surfaces for microwave-sintered sample and 87% tetragonal zirconia for conventional. The possible cause is that smaller grain size in microwave processing is helpful to avoid tetragonal zirconia to transfer monoclinic phase zirconia during cooling. More tetragonal zirconia in microwave-sintered sample would give a great contribution for transformation toughness.

The mechanical properties of Al_2O_3 sintered by microwave, however, are same as that by the conventional sintered samples. For instance, microwave

Table 1. Variation of grain size in sintered samples versus temperature

Temperature (°C)	Grain size for Al_2O_3[a] (µm)		Grain size for ZrO_2[b] (µm)	
	Microwave	Conventional	Microwave	Conventional
1000	0·40	0·40	—	—
1100	—	—	0·10	0·10
1150	0·50	0·42	—	—
1200	—	—	0·20	0·25
1300	1·15	0·65	0·32	0·36
1400	1·75	1·40	0·50	0·60
1500	2·54	2·47	0·60	0·88
1550	—	—	0·65	1·20

[a]Hold time: 30 min, heating and cooling rate: 20 °C min^{-1} for microwave process. Hold time: 120 min, heating and cooling rate: 3°C min^{-1} for conventional process.
[b]Hold time: 30 min, heating and cooling rate: 25°C min^{-1} for microwave process. Hold time: 120 min, heating and cooling rate: 3°C min^{-1} for conventional process.

Table 2. Property comparison for the samples sintered in microwave and conventional furnace

	Al_2O_3		$C–Y–ZrO_2$		PMZNT	
	Microwave	Conventional	Microwave	Conventional	Microwave	Conventional
Flexural strength (MPa)	$325 \pm 12\cdot2$	$331 \pm 10\cdot5$	$1200 \pm 22\cdot3$	$1160 \pm 21\cdot6$	$90 \pm 5\cdot2$	$65 \pm 4\cdot8$
Fracture toughness (MPa $m^{1/2}$)	—	—	$13\cdot7 \pm 0\cdot3$	$12\cdot6 \pm 0\cdot28$	—	—
Vickers hardness (GPa)	$18\cdot9 \pm 1\cdot5$	$18\cdot7 \pm 1\cdot2$	—	—	—	—
Breakdown strength (kV mm^{-1})	—	—	—	—	$105 \pm 6\cdot2$	$62 \pm 5\cdot8$

sintering and conventional sintering produced flexural strength of 325 and 331 MPa respectively. This is in good agreement with their microstructure where they had the same grain sizes. Similarly, comparable hardness, 18·9 and 18·7 GPa, between microwave and conventionally sintered samples was obtained. Patterson[25] also has reported comparable Young's modulus (398·5 and 397·1 GPa) and hardness (18·54 and 18·77 GPa) were observed in microwave and conventional methods for high purity alumina (>99·8%) to densities in excess of 98% of theoretical. All these results show that microwave sintering just produced comparable mechanical properties for the materials of Al_2O_3 with low dielectric loss.

4 Conclusions

Three kinds of ceramics with different dielectric loss (Al_2O_3, Ce–Y–ZrO_2 and PMZNT) were sintered to near theoretical density by multimode cavity with 2·45 GHz radiation. Thermal runaway and cracks of the samples were avoided by the controlling of input power and using of hybrid heating structure. Enhanced densification and shortened sintering time were observed for these materials, especially for high dielectric loss materials. Microwave sintering produced much finer and more uniform grain structure for lead-based relaxor ferroelectrics (PMZNT) which has strong dipolar losses, and Ce–Y–ZrO_2 which has higher ionic conductive loss compared to Al_2O_3. Grain sizes in samples sintered by microwave were reduced to half that of conventional sintering, such as from 6·8 to 3·2 µm for PMZNT and from to 1·2 to 0·65 µm for Ce–Y–ZrO_2. Their properties also were improved due to developed microstructure in microwave processing. Flexure strength and breakdown strength were increased from 65 MPa and 62 kV mm^{-1} to 90 MPa and 105 kV mm^{-1} for the relaxor ferroelectrics respectively. Fracture toughness and flexural strength reached to 13·7 MPa $m^{1/2}$ and 1200 MPa for Ce–Y–ZrO_2. However, microwave-sintered Al_2O_3 tends to have an equal grain size as conventional sintering. And this results in comparable flexural strength and hardness. Therefore, this work shows microstructure and properties of the ceramics sintered in a microwave furnace were significantly influenced by their dielectric loss mechanism.

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