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Effect of Y_2O_3 additive on conventional and microwave sintering of mullite

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Abstract

Mullite has become a strong candidate material for advanced structural and functional ceramics. Much interest has recently focused on sintering aids for mullite. The aim of this study was to evaluate the effect of Y_2O_3 as a sintering aid in the conventional and microwave sintering of mullite. To accomplish this study, a highly pure industrial mullite was used. Mullite with and without Y_2O_3 was pressed under a cold isostatic pressure of 200 MPa. Samples were sintered conventionally at 1400, 1450, 1500, 1550 and 1600 °C for 2 h and microwave-sintered for up to 40 min using a large range of power. The microstructure and physical properties of the microwave-sintered samples were compared to those of the conventionally sintered samples. The results showed that Y_2O_3 improved the densification of mullite bodies in the conventional and microwave sintering processes, but high densifications were achieved in just a few minutes when Y_2O_3 was used with microwave processing.

Keywords: A. Microwave process; D. Mullite; D. Y_2O_3; Sintering additives

1. Introduction

Mullite ($3Al_2O_3{\cdot}2SiO_2$) has become a promising candidate for high-temperature structural applications in an oxidizing atmosphere due to its unique combination of advantageous properties such as low thermal expansion, high creep resistance, superior thermal stability and excellent oxidation resistance [1–3]. However, mullite powder compacts have poor solid state sinterability because of the low interdiffusion rates of Si^{4+} and Al^{3+} within the mullite lattice [4].

Much interest has recently focused on mullite sintering aids [5–9] aimed at improving the sinterability of mullite compacts. Sintering additives usually promote the formation of liquid phase, which helps to reduce the viscosity of glassy (or liquid) phase [10] and decrease the mullite nucleation temperature in gel-derived powders [1,2], thereby leading to higher mobility of diffusing species. However, a number of reported additives such as MgO, La_2O_3, CeO_2, TiO_2, etc. promote anisotropic grain growth in mullite [11–14]. Reports in the literature involving studies of the influence of Y_2O_3 on mullite processing have focused on the nucleation of gel-derived mullites and on the conventional sintering of mullite compacts [15,16]. However, no investigations have been made to ascertain the influence of Y_2O_3 on the microwave sintering of mullite.

The properties of mullite bodies are determined by their microstructure and chemical composition, i.e., grain- and pore-size distribution, total porosity and grain-boundary characteristics. To control microstructural development and achieve the desired properties of the final product, parameters such as sintering temperature, soaking time and heating rate must be optimized [17]. Rapid heating has been reported to produce microstructural benefits, such as maintaining relatively fine microstructures compared with slow heating for similar densities and higher final densities [18,19]. Conventional fast firing, however, poses some difficulties. Differential sintering that causes differential densification is one of the problems most often encountered in conventional fast firing. In this context, microwave sintering has emerged in recent years as an alternative technique to overcome the problems of conventional fast firing. Because it is a noncontact technique, the heat is transferred to the product via electromagnetic waves and

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large amounts of heat can be transferred to material's interior, minimizing the effects of differential heating sintering [19].

There is growing interest in the application of microwave heating to sinter ceramics and metals [20–23]. Microwave sintering has many attractive features, including rapid volumetric heating, high production rates and low energy consumption [24–27]. In addition, it is believed [28–33] that the densification processes during sintering can be accelerated by microwave energy, achieving higher densities at lower sintering temperatures.

The purpose of this work is to evaluate the effect of Y_2O_3 as a mullite sintering aid on the conventional and microwave sintering process using a commercial mullite powder.

2. Experimental procedure

Commercial high purity (99.5%) mullite (SCIMAREC MP40) powder was used in this work. Pure mullite powder with and without Y_2O_3 was dispersed in an alcoholic medium by ball milling for 8 h. In the preparation of mullite with Y_2O_3, yttrium oxide (Sigma–Aldrich, 99.99%) was added in suitable amounts to obtain additive concentrations of 0.5 wt.%, 1.0 wt.% and 2.0 wt.% of Y_2O_3. The formulations containing 0.0 wt.%, 0.5 wt.%, 1.0 wt.% and 2.0 wt.% are identified here as Pure, 0.5Y, 1Y and 2Y, respectively. Disk-shaped samples (approximately 12 mm in diameter and 3 mm thick) were produced by unidirectional pressing under 40 MPa, followed by cold isostatic pressing (CIP) under 200 MPa. The average green density of the compacted disks was 58% of the theoretical density (3.17 g/cm^3), as determined from the dimensions and the weights. The green samples were sintered in a conventional furnace (Lindberg/Blue M Furnace) at 1400 °C, 1450 °C, 1500 °C, 1550 °C and 1600 °C with a soaking time of 120 min and a heating rate of 5 °C/min. The other samples were sintered in a microwave furnace (multimode cavity) at 2.45 GHz (Cober Electronics, MS6K) using susceptor materials as auxiliary heating elements. Details of the sintering assembly are given elsewhere [18]. Input power ranging from 0.9 to 2.4 kW and sintering times of up to 40 min were used in the microwave sintering processes. The cooling cycle was not controlled, but the entire microwave sintering (heating and cooling) cycle took <1 h.

The densities of the sintered samples were determined by the water-immersion technique using the Archimedes method. X-ray diffraction analysis was performed using CuKα radiation (40 kV and 40 mA as working conditions) (Siemens D-500) to identify the presence of Y_2O_3 related phases after the sintering process. Scanning electron microscopy (SEM) (PHILIPS, models XL30-FEG and XL30-TMP) was used to analyze the microstructural evolution. SEM samples were prepared according to the standard ceramographic techniques (cross-sections of the samples were polished and thermally etched). Grain sizes were measured using the linear-intercept technique and a stereographic correction factor of 1.56. At least 600 grains for each region were measured using the software Image-Pro Plus Program.

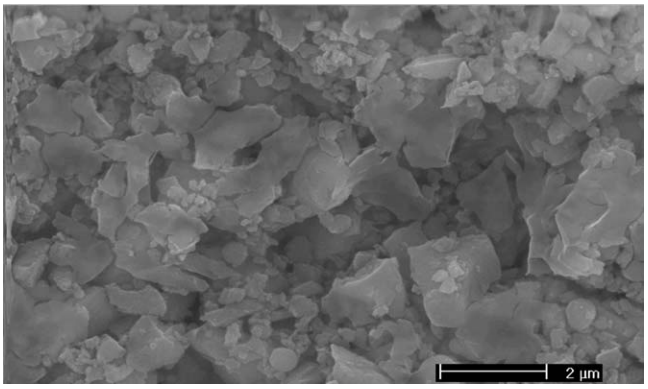

Fig. 1. SEM micrographs of mullite powder.

3. Results and discussion

A SEM micrograph of the mullite powder is depicted in Fig. 1. The mullite powder was composed of asymmetric plate particles and a few spherical particles. The particle sizes observed by SEM were consistent with the wide particle size distribution of this material (D_{10} and D_{90} of about 0.7 μm and 3.0 μm, respectively).

The densities of pure mullite and conventionally sintered mullite with Y_2O_3 additive are shown in Fig. 2. The density of the pure mullite after sintering at 1400 °C was almost equal to the green density (58%). At 1400 °C, the addition of 0.5 wt.% and 1.0 wt.% of Y_2O_3 increased the density up to only 64% and 67%, respectively; however, the addition of 2 wt.% increased the density to 78%. A similar behavior was observed at 1450 °C, but a more pronounced influence of Y_2O_3 on the improvement of the final density was observed at 1500 °C. The addition of 0.5 wt.% increased the relative density to 87%, while the addition of 1 wt.% and 2 wt.% of Y_2O_3 increased the density to 92% and 95%, respectively. At 1550 and 1600 °C, additions of only 0.5% increased the density to values of around 95%, but no significant differences in density occurred in response to the increase of Y_2O_3 from 0.5 to 2.0%. These

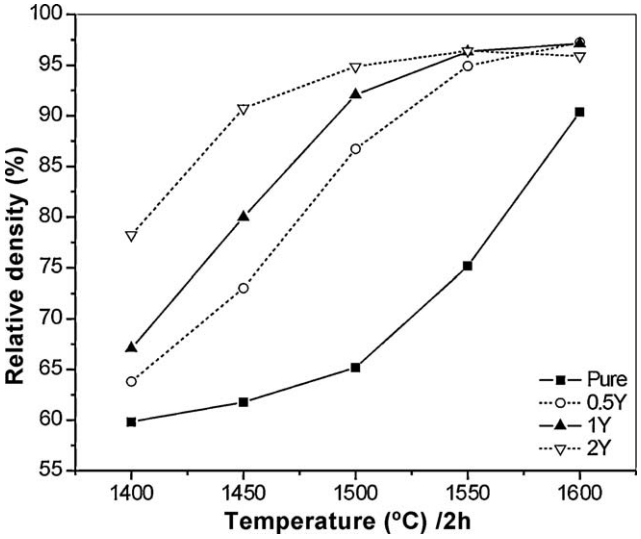

Fig. 2. Relative density of conventionally sintered samples.

results indicate that sintering temperatures above 1550 °C would not have effectively improved the densities of the Y_2O_3-additived mullite bodies studied here.

It is evident that Y_2O_3 favors the densification of mullite bodies. At all the temperatures tested here, the pure mullite presented lower final densities, which is consistent with reports in the literature [2,9,10,16]. The higher densities of the mullite bodies were attributed to the formation of a liquid of low viscosity resulting from the addition of the additive.

Fig. 3 shows the densities of the microwave sintered mullite with Y_2O_3. At power levels of 0.9, 1.2 and 1.5 kW and a sintering time of 20 min, the densities of pure and with Y_2O_3 samples were not significantly increased. This may be ascribed to insufficient energy (power and/or time) to increase the temperature of the samples and the development of a sintering process using microwave energy. However, at power levels exceeding 1.5 kW (for 20 min), the final relative density increased with Y_2O_3 content and the samples reached higher densities than the pure ones. Samples with 2% of Y_2O_3 and sintered at 2.4 kW achieved densities of approximately 95%. The pure samples showed practically no increase in density, despite the increase in the power level to 2.4 kW with only 20 min of sintering.

The application of power levels of 0.9 and 1.2 kW and 30 min sintering in the microwave sintering process did not improve the densities of pure or with Y_2O_3 samples. However, at 1.5 kW, the 2Y samples' densities increased to about 77%. This power level and sintering time appears to be insufficient for pure mullite to reach the critical temperature and to interact with and absorb microwaves efficiently, since the relative density did not increase significantly. When higher power levels were used, the pure and with additive samples showed markedly higher relative densities. However, the samples with Y_2O_3 presented higher densities than the pure mullite. At 1.8 kW the pure mullite reached 73% after 30 min of sintering, while the 0.5Y, 1Y and 2Y samples achieved densities of 85, 91 and 93%, respectively. The densities of the 1Y and 2Y samples were very similar from 1.8 to 2.4 kW, unlike what was observed after sintering for 20 min, indicating that it is possible to achieve high densities with low amounts of Y_2O_3 using longer sintering times in the microwave processing.

At a sintering time of 40 min and power levels of 0.9 and 1.2 kW, the sintering behavior was similar to that shown during sintering for 30 min. On the other hand, when power level was increased to 1.5 kW, the 2Y sample reached a density of 90%. Increasing the power level led to very similar densities in the samples with Y_2O_3, and at 2.1 and 2.4 kW, the samples with Y_2O_3 reached a density of approximately 95%. Mullite does not absorb microwave energy efficiently at room temperature and poor microwave absorption characteristics make its initial heating difficult, despite the use of high power levels. On the other hand, Y_2O_3 promotes liquid phase formation, and the development of a liquid phase increases the body's dielectric loss and consequently its microwave absorption characteristic. Thus, it is assumed that the higher densities of the doped bodies are attributable not only to the development of a low viscosity liquid phase in the microwave processing but also to the influence of this phase on the absorption characteristics of the body.

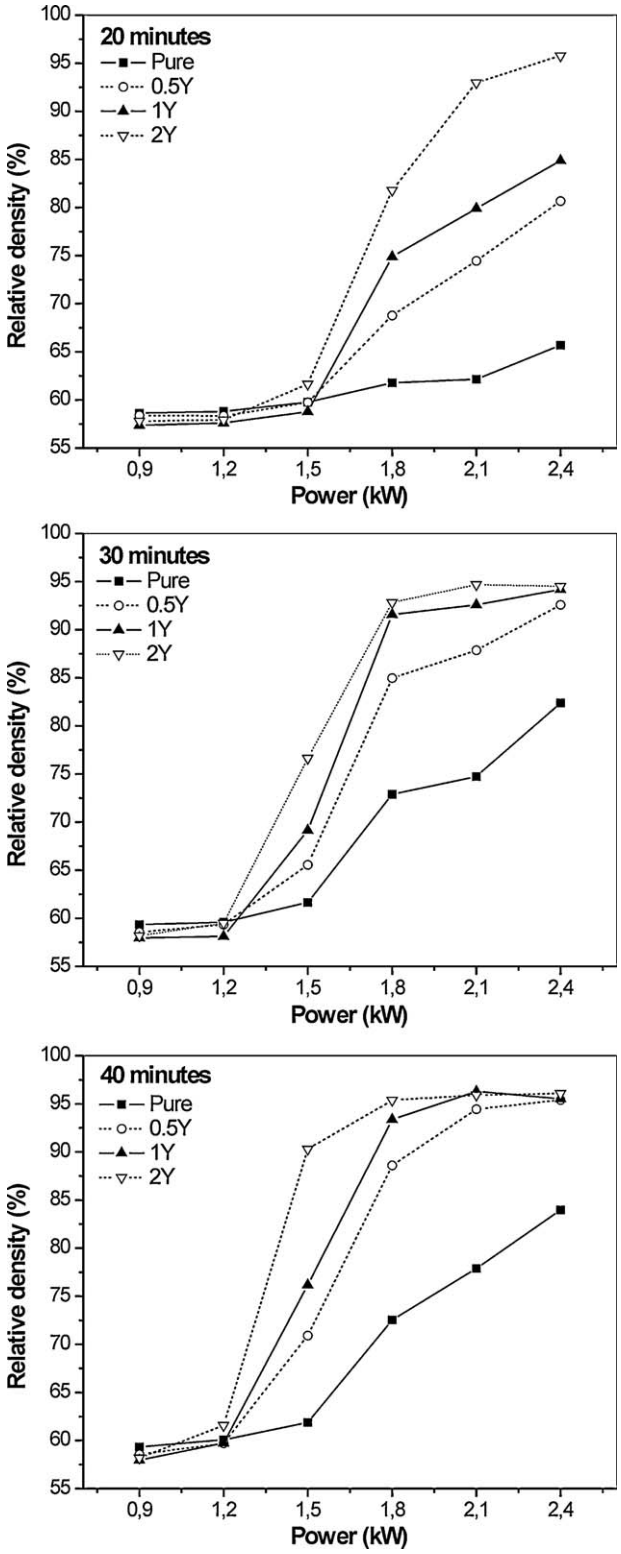

Fig. 3. Relative density of microwave sintered samples for 20 min, 30 min and 40 min.

Fig. 4 shows the XDR pattern of 2Y samples conventionally sintered, while Fig. 5 presents the XDR pattern of pure, 0.5Y, 1Y and 2Y samples microwave-sintered at 2.4 kW/40 min.

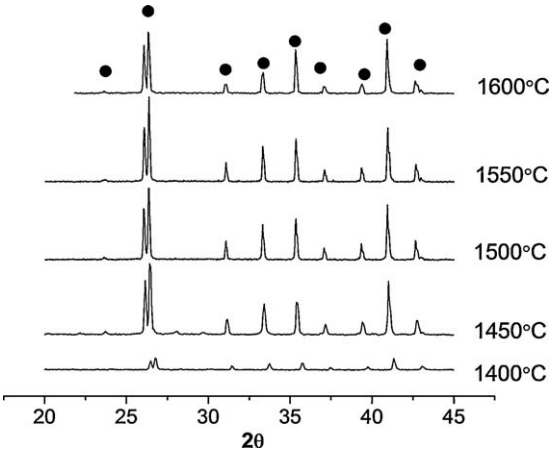

Fig. 4. XRD patterns of conventionally sintered 2Y sample (●: mullite).

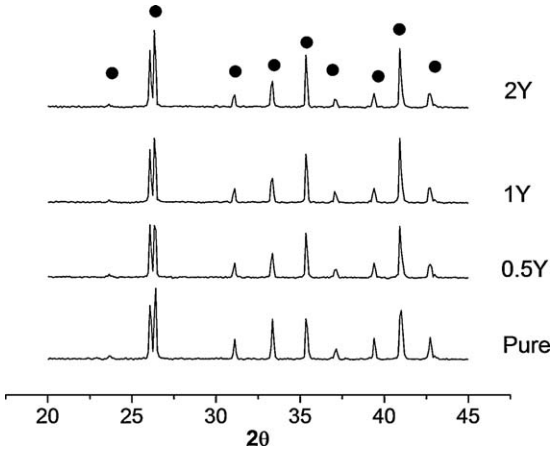

Fig. 5. XRD patterns of pure, 0.5Y, 1Y and 2Y samples microwave-sintered at 2.4 kW for 40 min (●: mullite).

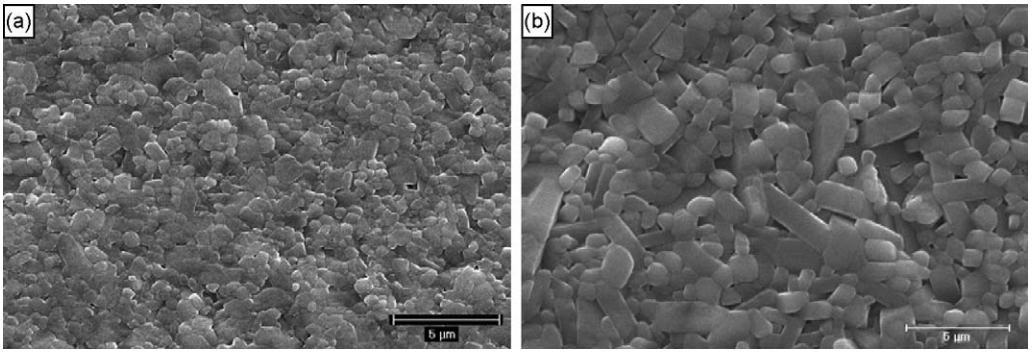

Fig. 6. SEM micrographs of samples conventionally sintered at 1500 °C/2 h (a) Pure and (b) 2Y.

Unlike other researches [1,2,16], no peaks associated to the addition of Y_2O_3, such as $Y_2Si_2O_7$ or Y_2SiO_5, were observed. The amount of Y_2O_3 used was probably too small to favor the formation of secondary phases.

Figs. 6–8 show SEM micrographs of conventionally sintered samples. The addition of Y_2O_3 favored grain growth, and the samples with Y_2O_3 exhibited a larger average grain size. However, a comparison of the densities of pure and Y_2O_3 doped samples revealed a negligible increase in average grain size of the samples with additions of Y_2O_3. At 1500 and 1550 °C, the 2Y samples presented wider grain size distribution, with elongated grains. After firing at 1600 °C, all the samples with Y_2O_3 displayed heterogeneous microstructures due to the development of elongated grains.

Figs. 9 and 10 show SEM micrographs of microwave-sintered samples. The 0.5Y and 1Y samples microwave-sintered for 30 min presented homogeneous structures after sintering at 1.8 and 2.1 kW, while the micrograph of the 2Y sample shows elongated grains at these power levels. When 2.4 kW was applied, the 1Y and 2Y presented elongated grains

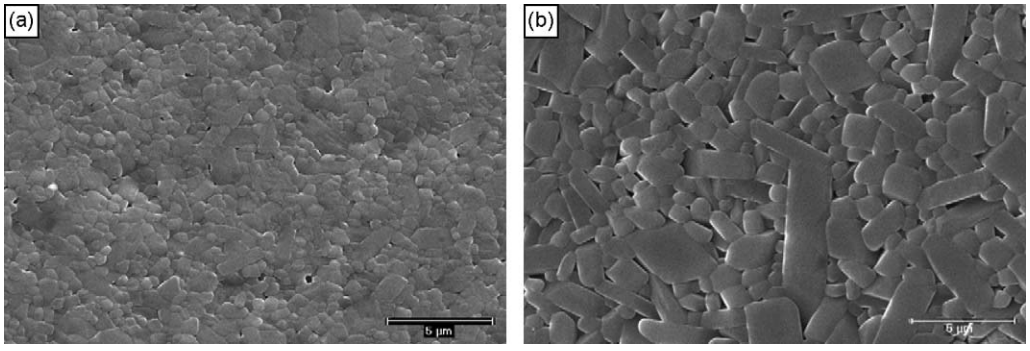

Fig. 7. SEM micrographs of samples conventionally sintered at 1550 °C/2 h (a) pure and (b) 2Y.

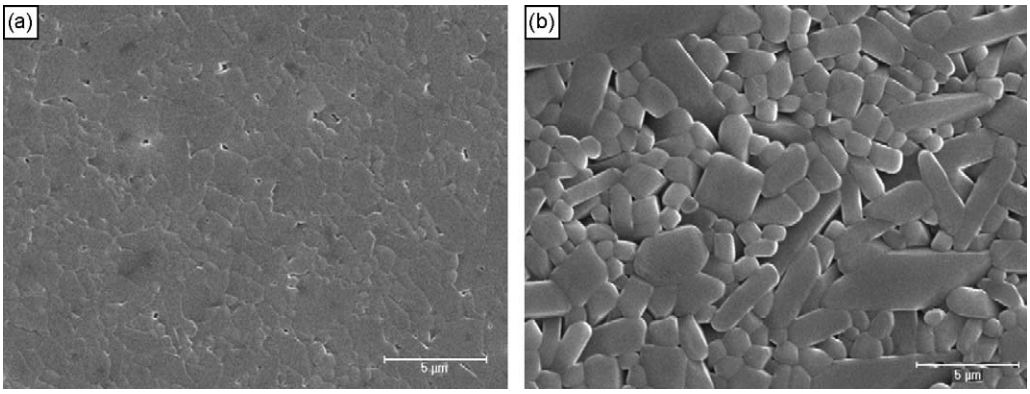

Fig. 8. SEM micrographs of samples conventionally sintered at 1600 °C/2 h (a) pure and (b) 2Y.

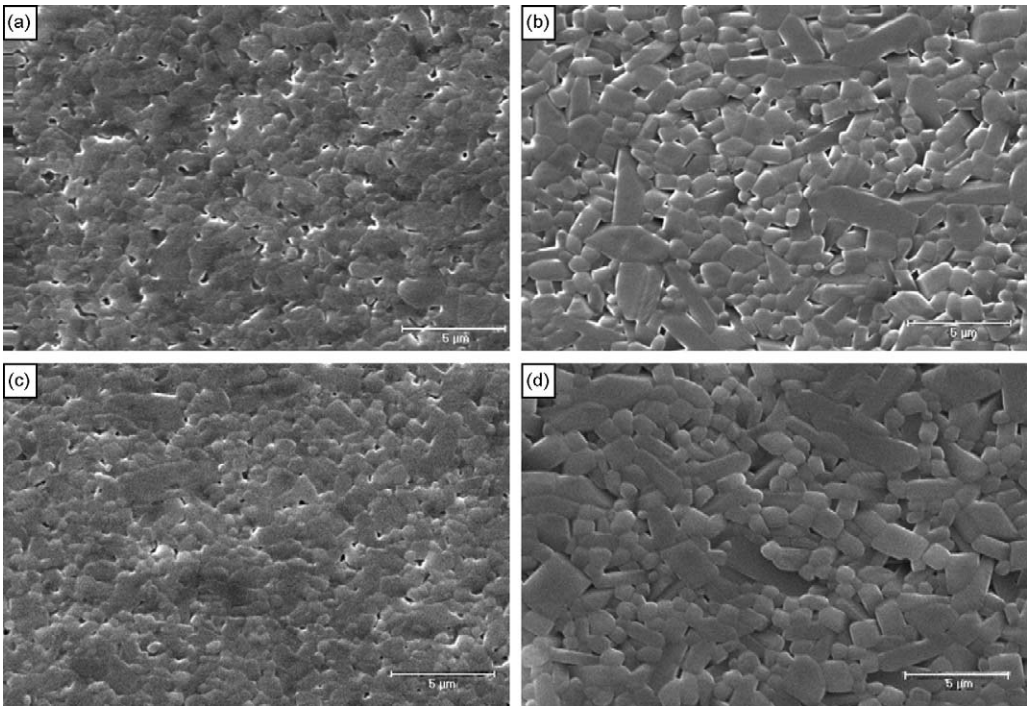

Fig. 9. SEM micrographs of samples microwave-sintered at: 2.1 kW/30 min (a) pure, (b) 2Y; 2.4 kW/30 min (c) pure, (d) 2Y.

and only the 0.5Y had a homogenous microstructure. This behavior may be associated with the amount of liquid phase developed during microwave sintering. Use of 2% of Y_2O_3 led to the formation of a larger amount of liquid phase, enhancing the absorption of microwaves, promoting a rise in temperature and accelerating diffusion. These conditions favored not only densification but also grain growth and the formation of elongated grains. Thus, lower power levels are required to sinter 2Y samples without the development of elongated grains.

The 0.5Y and 1Y samples presented lower Y_2O_3 content and consequently the amount of liquid phase generated was lower. Grain elongation was not observed after sintering at 1.8 and 2.1 kW, despite the high densities achieved by the 1Y sample. It is interesting to note that the 0.5Y sample did not reach high densities after sintering at 1.8 and 2.1 kW. This suggests that a balance is needed between the amount of additive and the power level in the microwave sintering process to attain not only high densities but also homogeneous microstructures.

The doped samples microwave-sintered for 40 min presented a microstructural evolution similar to those sintered for 30 min. A power level of 1.8 kW for 40 min applied to the 0.5Y sample did not lead to elongated grains, but the 1Y and 2Y presented elongated grains. At a sintering time of 30 min and power level of 1.8 kW applied to the doped samples did not result in elongated grains in 1Y sample.

The micrograph of the 0.5Y sample sintered at 2.1 kW for 40 min indicated the initial development of elongated grains. The 1Y and 2Y samples showed elongated grains at 1.8 kW for 40 min, but marked growth of this type of grain was not observed at 2.1 kW, and the average grain size of these samples was practically the same, as indicated in Table 1. The 0.5Y and 1Y samples sintered at 2.4 kW presented heterogeneous microstructures due to the presence of elongated grains. In this

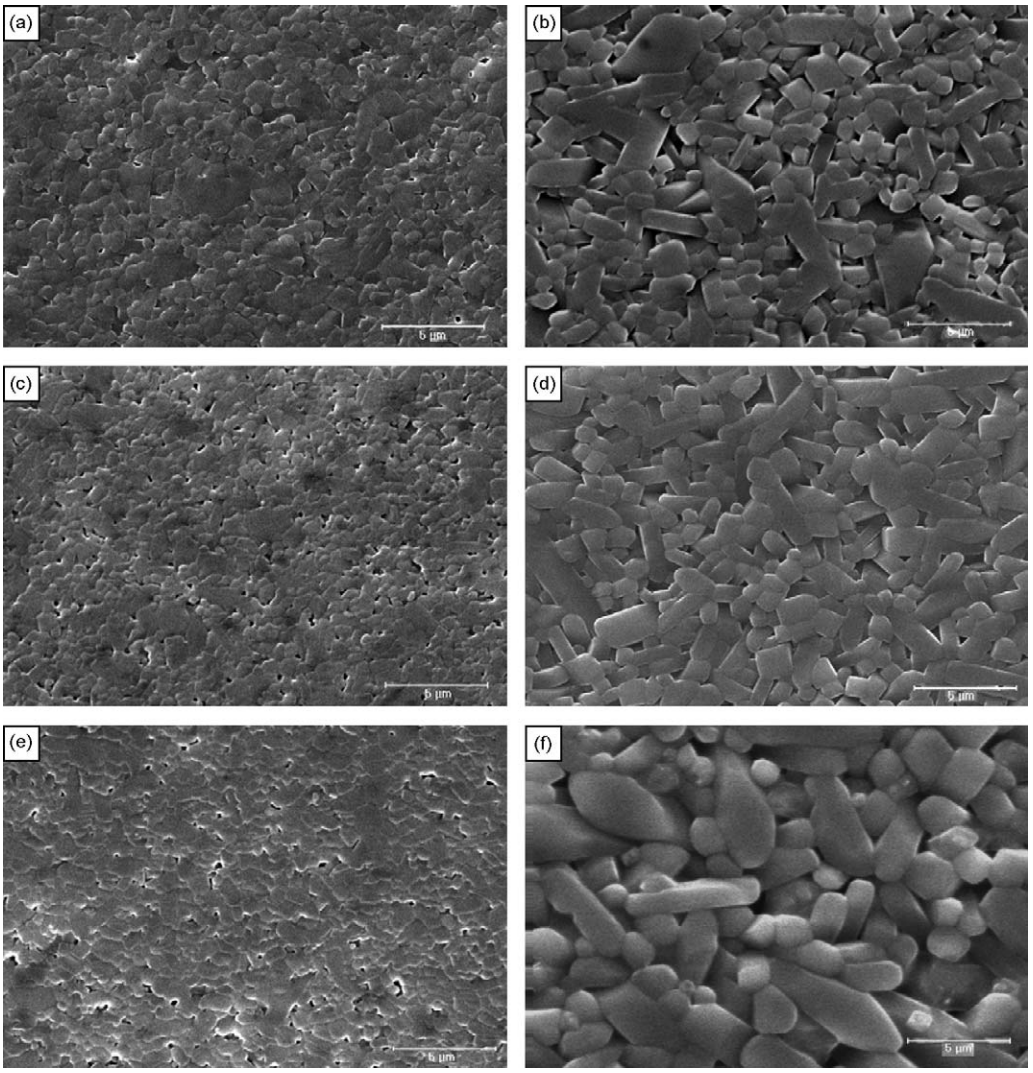

Fig. 10. SEM micrographs of samples microwave-sintered at: 1.8 kW/40 min (a) pure, (b) 2Y; 2.1 kW/40 min (c) pure, (d) 2Y; 2.4 kW/40 min (e) pure, (f) 2Y.

Table 1
Average grain size of conventionally and microwave-sintered samples.

Sintering condition		Average grain size (μm)			
		Pure	0.5Y	1Y	2Y
Conventional	1500 °C/2 h	1.05 ± 0.52	1.84 ± 1.19	2.06 ± 1.36	2.23 ± 1.38
	1550 °C/2 h	1.09 ± 0.56	1.82 ± 0.95	2.11 ± 1.08	2.15 ± 1.16
	1600 °C/2 h	1.94 ± 1.18	2.74 ± 1.77	2.65 ± 1.80	2.73 ± 1.66
Microwave	1.8 kW/40 min	1.38 ± 0.73	1.75 ± 0.88	1.93 ± 1.02	1.97 ± 1.15
	2.1 kW/40 min	1.30 ± 0.76	2.01 ± 1.27	2.07 ± 1.28	2.28 ± 1.35
	2.4 kW/40 min	1.29 ± 0.65	1.82 ± 0.90	2.11 ± 1.25	2.49 ± 1.57

condition, the 2Y sample presented the coarser microstructure. The condition of 2.4 kW and 40 min of sintering favored grain growth in the 2Y sample to the point where microstructure was not heterogeneous but showed basically elongated mullite grains.

A comparison of the microstructures and average grain sizes of the pure and doped microwave-sintered mullite revealed that, in every condition, the pure samples presented a more porous microstructure and lower average grain sizes. It was also found that the pure mullite did not show elongated grains in any of the conditions of microwave sintering. Our results indicated that microwave-sintered samples had more homogeneous microstructures than conventionally sintered samples, in a comparison of similar densities. The average grain size of the microwave-sintered samples was smaller than that of conventionally sintered samples, probably due to rapid firing and hence shorter sintering cycles, which reduce grain growth. In the microwave sintering of

mullite, the amount of Y_2O_3 can be different from that used in conventional sintering, depending on power level and sintering time (amount of energy available for the process). This is because Y_2O_3 strongly affects the microwave absorption characteristics of mullite, so that densification and grain size behavior are closely related to the parameters of additive content and available energy. Another important observation is the homogenous distribution of Y_2O_3 in the mullite bodies (after sintering, Y_2O_3 particles and grains or pockets of liquid phase were not observed by backscattered SEM), despite its coarse grain size and the short sintering cycle. This may be due to specific heating of the additive and the accelerated diffusion process promoted by microwave energy. Many investigators have reported unexpected effects resulting from the use of microwave radiation as an alternative energy source during the processing of materials. These results have included apparent evidence of accelerated kinetics for a range of processes in ceramic, polymeric and organic systems, and enhanced sintering of ceramic powder compacts, including lower sintering temperatures. These unexpected effects are called the ''microwave effect''. As a general summary, the kinetics of synthesis and sintering reactions are reportedly augmented by 2 or 3 orders of magnitude. Thus, the homogenous dispersion of Y_2O_3 in the mullite bodies may be associated with specific heating and the ''microwave effect''.

4. Conclusions

The effect of Y_2O_3 additive on rapid microwave sintering of mullite was investigated here. Y_2O_3 exerted a stronger influence on the densification process in microwave sintering than in conventional sintering. The addition of Y_2O_3 favored grain growth, which was proportional to the amount of dopant, but a comparison of the same densities revealed that the grain sizes of microwave-processed samples were smaller. Using microwave energy, it is possible to use a coarse additive in the rapid sintering of mullite while maintaining its homogeneous distribution in the body. The microwave energy associated with Y_2O_3 additive drastically reduced the processing time and produced high density bodies.

Acknowledgements

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