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Abstract

Better understanding of the effect of multimode-microwave sintering of zirconia-toughened alumina (ZTA) was investigated. A comparative dilatometric analysis was conducted between conventional and microwave heating processes, to clarify the influence of zirconia on the densification of ZTA under electromagnetic field. The thermal gradient on sample measurements indicates the change to the microwave volumetric heating is improved by zirconia which adsorbs microwave energy better, thus acting as a susceptor. The most beneficial effect on microstructure, toughness, and hardness were observed at the optimal zirconia content of 10 vol%. The results with both microwave and conventional sintering illustrate the strengthening effect on the composite by zirconia. Of special interest, multimode microwave sintering creates a finer homogeneous microstructure, with resulting hardness and toughening comparable to those obtained for conventional sintering, as well as improved densification, and at lower cost.

KEYWORDS
alumina, Field Assisted Sintering Technology (FAST), microwaves, zirconia

1 | INTRODUCTION

For many years, reducing the environmental impact of industrial processes has been the focal point of research. Since ceramics sintering is energy intensive, utilizing microwave heating is a promising alternative for a more ecofriendly industry. Evidently, the potential savings in money could be advantageous for companies as well.

In conventional processing, ceramics are baked in ovens via electrical resistances with thermal conduction, convection and radiation. Unfortunately, this process requires heating the entire space in the kiln and takes a lot of time to adequately sinter the ceramic. While, microwave can target only or almost only the matter: the ceramic material has a direct interaction with the microwave field depending on its dielectric properties. This offers several advantages over conventional methods,[1] for example, high heating rate, lower sintering temperature, enhanced diffusion for densification, finer and/or uniform microstructure, potential optimized final properties of the sample, etc. Several scientific articles discussed the beneficial effect of microwave on the sintering of ceramics,[2] note: the "Ponderomotive Force"[3] is usually considered as the probable cause.

Consequently, many kinds of ceramics can be treated at high temperature using this more efficient and innovative heating method. Our work focuses on ceramic composites of Al_2O_3–ZrO_2.

1.1 | Conventional sintering of Al_2O_3–ZrO_2

Al_2O_3–ZrO_2 composites (otherwise known as Zirconia-Toughened Alumina: ZTA) have been widely investigated in numerous publications. This composite has the advantages of both: hardening and chemical stability of alumina, and the toughening by phase transformation of zirconia. The concept of ZTA is to disperse a volume of tetragonal

zirconia in the alumina matrix (generally up to 20 vol%). Thus, zirconia can improve the fracture toughness of the matrix due to the tetragonal/monoclinic phase transformation. Different mechanisms controlling the toughening within ZTA have been discussed in detail in literature. The first is stress-induced transformation toughening: under external tensile stress, the metastable tetragonal zirconia phase will transform to a stable monoclinic phase; this is followed by a volume expansion (~4%) and shear strain (~6%) which can provide a compressive stress leading to a reduction and finally a stop in crack propagation.[4] The second is due to "microcracks" in the composite: when the phase transformation appears during the cooling after sintering temperature, microcracks occur inside the material (mainly caused by the volume expansion). These microcracks can absorb fracture energy, and consequently improve strongly the mechanical properties of composite. For example, the toughness could reach 7 MPa $m^{1/2}$ for an optimal fraction of zirconia. In consequence, ZTA is widely used in manufacturing of cutting tools, dies, or prosthesis components.[5] Notice that many studies have mentioned a delay effect of zirconia on sintering of ZTA composites.[6]

1.2 | Microwave sintering of Al_2O_3–ZrO_2

Some authors have demonstrated the feasibility of firing ZTA composites using microwave.[7,8] Compared with conventional methods, the microwave hybrid heating on ZTA reaches almost full density in a shorter time cycle and at a lower sintering temperature. Furthermore, microwave heating is a good way to suppress the grain growth of ZTA and to provide a more uniform microstructure Binner et al.[9] attest that 3YSZ nanopowders fired by microwave have a finer grain size than using conventional process. Consequently the mechanical properties of materials are enhanced. Furthermore, Benavente et al.[7] have shown that ZTA composites sintered by microwave have higher density, hardness and young's modulus in comparison to conventional heating. These previous works only centered on the analysis within final sintered samples, for example, the relation between microstructure and mechanical properties of singlemode-microwave-sintered ZTA composites.[7] They did not yield any information about dilatometric curves or microwave coupling behaviors during the whole heating process under an electromagnetic field. In addition, applying identical thermal cycles for both conventional and microwave heating processes was usually ignored.[7] Obviously, an identical thermal cycle should be meaningful to compare the effect of heating methods on ZTA composite microstructure and properties.

Therefore, systematically and comparatively studying conventional and microwave sintering of ZTA composites is our objective. To broadly examine the potential effect of microwave on the densification of ZTA, it seems meaningful to investigate the influence of zirconia amount on microwave process due to its different dielectric property compared with alumina. The microwave coupling capability of materials depends on their dielectric properties: coupling increases when the dielectric loss is more significant. Dielectric loss quantifies the dissipation of electromagnetic energy in a dielectric material, and is defined as follows: $\varepsilon^*=\varepsilon'-j\varepsilon''$ where ε^* is called complex permittivity. The dielectric dissipation factor is called tanδ with: $\tan\delta=\varepsilon''/\varepsilon'$. The more the dielectric material has a high value of ε'', the more it can be heated by microwave. Dielectric properties of ceramics at high temperature, recorded at 2.45 GHz, were studied by Aral et al.[10] They found that the complex permittivity at 600°C is, respectively, $\varepsilon^*=20.5-2j$ (ie, $\tan\delta \approx 0.09$) for zirconia and $\varepsilon^*=9.7-0.081j$ (ie, $\tan\delta \approx 0.008$) for alumina. These data showed that zirconia couples much more with microwave than alumina. The electromagnetic field should have different effects on these two phases within ZTA. Specific, in situ, contactless optical dilatometry and thermal gradient were used for analysis. Then measuring the mechanical properties of both alumina and ZTA composites and analyzing the relationship between microstructure and properties were conducted. On the basis of this research, we tried to show the possible differences in densification behavior as well as in mechanical properties between conventionally sintered and multimode microwave-sintered Al_2O_3–ZrO_2 composites with different amounts of ZrO_2.

2 | EXPERIMENTAL PROCEDURE

2.1 | Starting powders and consolidation of green samples

Three reference compositions were used: 97 vol% alumina—3 vol% zirconia, 90 vol% alumina—10 vol% zirconia, and 60 vol% alumina—40 vol% zirconia (ZTA97, ZTA90 and ZTA60, respectively, Baikowski International, La Balme-de-Sillingy, France). These alumina-zirconia powders were obtained by mixing two pure (<220 ppm of impurities) and very fine powders. We also used α-alumina powders (BA15, 15 m^2/g; Baikowski International) without dopants, but containing 2 wt% of binders and plasticizer.

Starting powders and the microstructure of sintered samples were observed by Scanning Electron Microscopy (SUPRA 55, Carl Zeiss, Oberkochen, Germany). In Figure 1, the SEM micrograph of ZTA90 powders shows spherical spray dried granules with a diameter between 10 and 50 μm. Otherwise, the granules seem to be dense which could be very advantageous when improving the mechanical resistance of green sample.

Two different sized steel dies were employed. For the microwave methods cylindrical samples were shaped by uniaxial compressing at 50 MPa in a 12 mm die to facilitate the precision of temperature measurement and thermal gradient evaluation on sample. An 8 mm die was used for conventional sintering to adapt to the size requirement of dilatometer. To have higher homogeneity and isotropy stress in green samples, they were both then formed by cold isostatic pressing at 300 MPa. Any organic compound was removed by heating at 2°C/min to 600°C with a dwell time of 1 hour in air. Green densities were determined by measuring the sample dimensions and weight. The average values obtained through three samples measurements were between 50.6 ± 0.4% and 51.9 ± 0.4% of theoretical density (T.D.), as shown in Table 1. Densities were slightly lower in the case of samples for microwave sintering with a diameter of 12 mm but within experimental limits.

2.2 | Conventional and microwave heating systems

Conventional sintering experiments were performed in air using a dilatometer (Setsys 16/18, Setaram, Caluire, France). Temperature measurements were monitored with an S-type thermocouple. The microwave sintering system consisted of a fixed frequency of 2.45 GHz microwave generator (GMP30K; Sairem, Neyron, France)with adjustable output power (~3 kW), and connected to a multimode cavity with an inside dimension of 430 mm × 430 mm × 490 mm. Due to the low dielectric loss factor (tanδ) of the materials at low temperature, a SiC ring was used to initiate sample heating up to the temperature where material could adequaly absorb microwave. The same configuration was described by Zuo et al.[11] Refractory materials transparent to microwave were used. Because of the electromagnetic field inside the cavity, in situ dilatometric measurement had to be implemented with a contactless and external device to avoid interference and measurement errors. This experimental system combines a high-resolution CCD camera (SLC2050MTLGEC; 14-bit, 1600 × 1200, SVS-VISTEK, Germany), and two infrared pyrometers with one or two colors (5G-1007 and 5R-3015, IRCON, Santa Cruz, CA, USA). Pyrometer temperature was calibrated using metals with well-known melting points. The contactless measurement system and temperature calibration procedure were described by Żymełka et al.[12] and Zuo et al.[2] This contactless method precisely controls the thermal cycle and so compared microwave and conventional heating processes rigorously. For each composition, three identical tests were run, to furnish superimposable curves. Temperature distribution on sample during microwave sintering was also carried out by linking the CCD camera with two pyrometers. The gray levels of recorded images were converted into a temperature field.

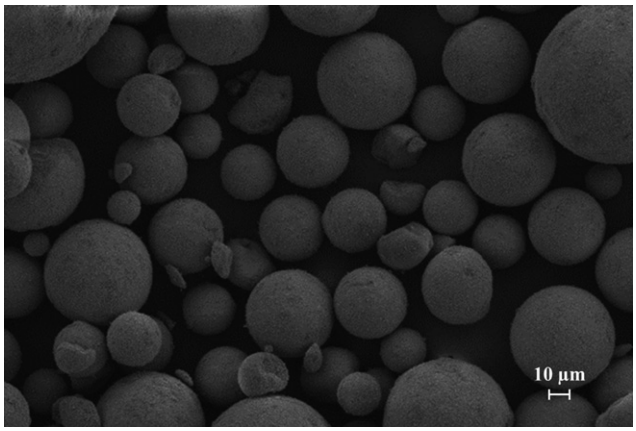

FIGURE 1 SEM micrograph of initial ZTA90 powders

TABLE 1 Parameters for each composition of green samples

	Sample diameter (mm)	Thickness (mm)	Theoretical density (g/cm^3)	Heating mode	ρ_0 (% T.D.)
BA15	8	7.1	3.987	CS	51.8
	12	4.8		MW	50.7
ZTA97	8	6.6	4.010	CS	51.8
	12	4.3		MW	51.7
ZTA90	8	6.7	4.160	CS	51.7
	12	4.5		MW	50.6
ZTA60	8	5.9	4.810	CS	51.9
	12	4.1		MW	50.8

2.3 | Characterization

Densities and the rate of densification were deduced from the final densities measured by Archimedes method with ethanol the immersion fluid as well as from the recorded shrinkage data. Final density was averaged from at least three measurements and the accuracy is about ±0.3%. Average grain size of sintered alumina sample was evaluated with SEM micrographs of polished sample on at least 200 grains using intercept method. A statistical correction factor of 1.56 was applied to the apparent grain size measured.[13]

Mechanical characterizations were conducted with a micro Vickers hardness tester (MTX70, Matsuzawa, Akitashi, Japan). To measure hardness, a load of 500 g was applied during 15 seconds (15 tests/sample). The fracture toughness was determined by the indentation method on polished specimens, as explained by Liang et al.[14] The equation below was used to evaluate the fracture toughness, where Φ is a constant (equal to 3), H the hardness, E the Young's modulus, α a coefficient as a function of

Poisson's ratio, a the diagonal of indent, and c the crack radius. E and Poisson's ratio for each composition are determined using mixture law from the pure Al_2O_3 and ZrO_2 values.

$$\left(\frac{K1C.\Phi}{H.a^{1/2}}\right).\left(\frac{H}{E.\Phi}\right)^{0.4}.\alpha = \left(\frac{c}{a}\right)^{(c/18a)-1.51}$$

This method measures the size of cracks formed around the indent. Indentation experiments used loads of 3, 5, or 10 kg (15 tests/sample). Before measurement, the specimens were annealed (conventional heating at 1200°C, 15 minute) to remove surface compressive stresses.

3 | RESULTS AND DISCUSSION

3.1 | Densification behaviors

As aforementioned to better compare the influence of microwave on densification, the same thermal cycle was applied in both conventional and microwave heating. The heating rate was 25°C/min up to 1500°C. After reaching maximal temperature, the generator was switched off to obtain a rapid and natural thermal mass cooling. Figure 2 shows the densification behavior during conventional and microwave sintering of alumina and ZTA composites. It is clearly seen that alumina shrinkage starts at 1000°C for both sintering techniques. In the case of ZTA composites, the shrinkage starts at higher temperature (between 1100°C and 1200°C), indicating that the addition of zirconia delays the densification process.

According to Figure 2A, microwave effect appears at the intermediate stage of sintering: at a given temperature, interestingly the densities of all the samples under microwave heating are higher than those under conventional heating, whatever the zirconia content.

To better show the microwave enhancement on densification, curves of densification rate over temperature have been plotted for samples sintered at a heating rate of 25°C/min. According to Figure 2B, densification rate is higher for composites containing zirconia. Starting from these data, Figure 3 shows the evolution of the temperature for which the maximum densification rate was observed as a function of zirconia content. On the one hand, it can be seen that when zirconia was added, the maximum densification rate appeared at a higher temperature. It should be associated with the delay effect of zirconia on sintering of ZTA, as described previously. On the other hand, the microwave field shifts the temperature corresponding to the maximum densification rate toward lower temperatures.

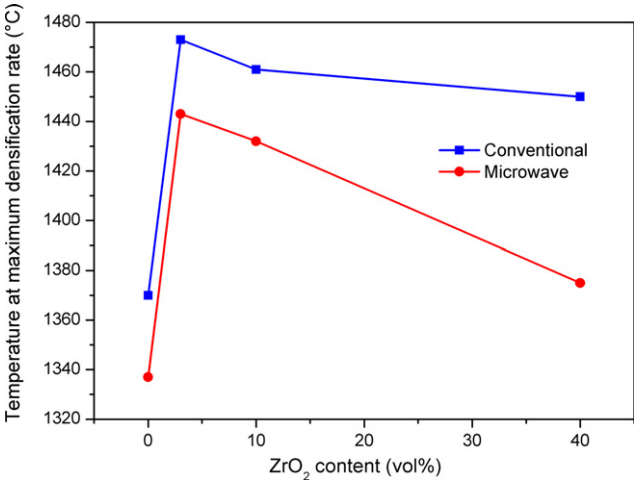

FIGURE 3 Evolution of temperature at maximum densification rate as a function of zirconia content [Color figure can be viewed at wileyonlinelibrary.com]

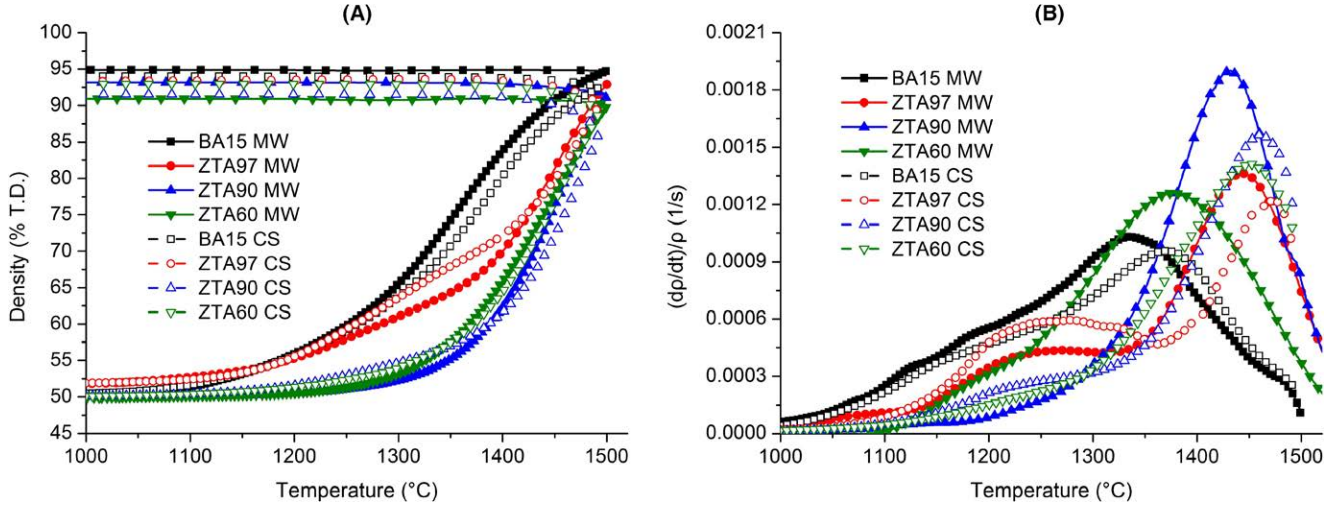

FIGURE 2 Comparison of densification behaviors between microwave (MW) and conventional (CS) sintering of alumina and ZTA: (A) relative density vs temperature; (B) densification rate vs temperature [Color figure can be viewed at wileyonlinelibrary.com]

This confirms the positive effect of the electromagnetic field on densification. As shown in Figure 2A, for ZTA60, the density curves intersect during cooling due to a slower rate in conventional dilatometer than in microwave system. Conventionally, the sintering continues for a longer time after reaching the maximum temperature. So, to evaluate both processes equally, densities were plotted at 1500°C vs the zirconia content in Figure 4.

Conventional sintering: Figure 4 confirms again the effect of zirconia, which retards alumina matrix densification. The strongest difference between microwave and conventional heating was observed with content around 10% (ZTA90). The percolation threshold of zirconia is reached for ZTA60, and the zirconia sintering occurs, which enhances the densification of composite.

Microwave sintering: therefore, microwave heating process could bring about higher final densities compare to standard methods, independently of the zirconia content. The gap between the densities of conventionally sintered and microwave-sintered samples was narrow (~2%) for pure alumina and ZTA60, but increased up to 6% in the case of ZTA90. It was observed that zirconia addition delays the densification of conventionally sintered alumina, especially for the ZTA90 composition. However, the final density of ZTA90 is surprising much higher. This result shows that even at zero zirconia, the microwave effect on alumina sintering is significant, but especially dramatic for ZTA90. Note that from ZTA 90 just until ZTA 60 the

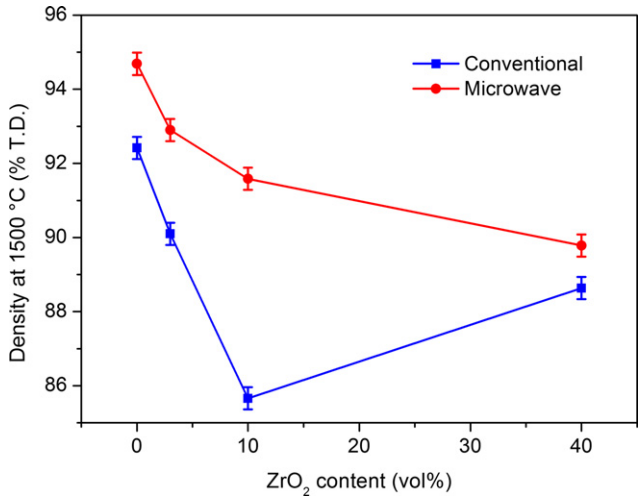

FIGURE 4 Density at 1500°C as a function of zirconia content for conventional and microwave sintering [Color figure can be viewed at wileyonlinelibrary.com]

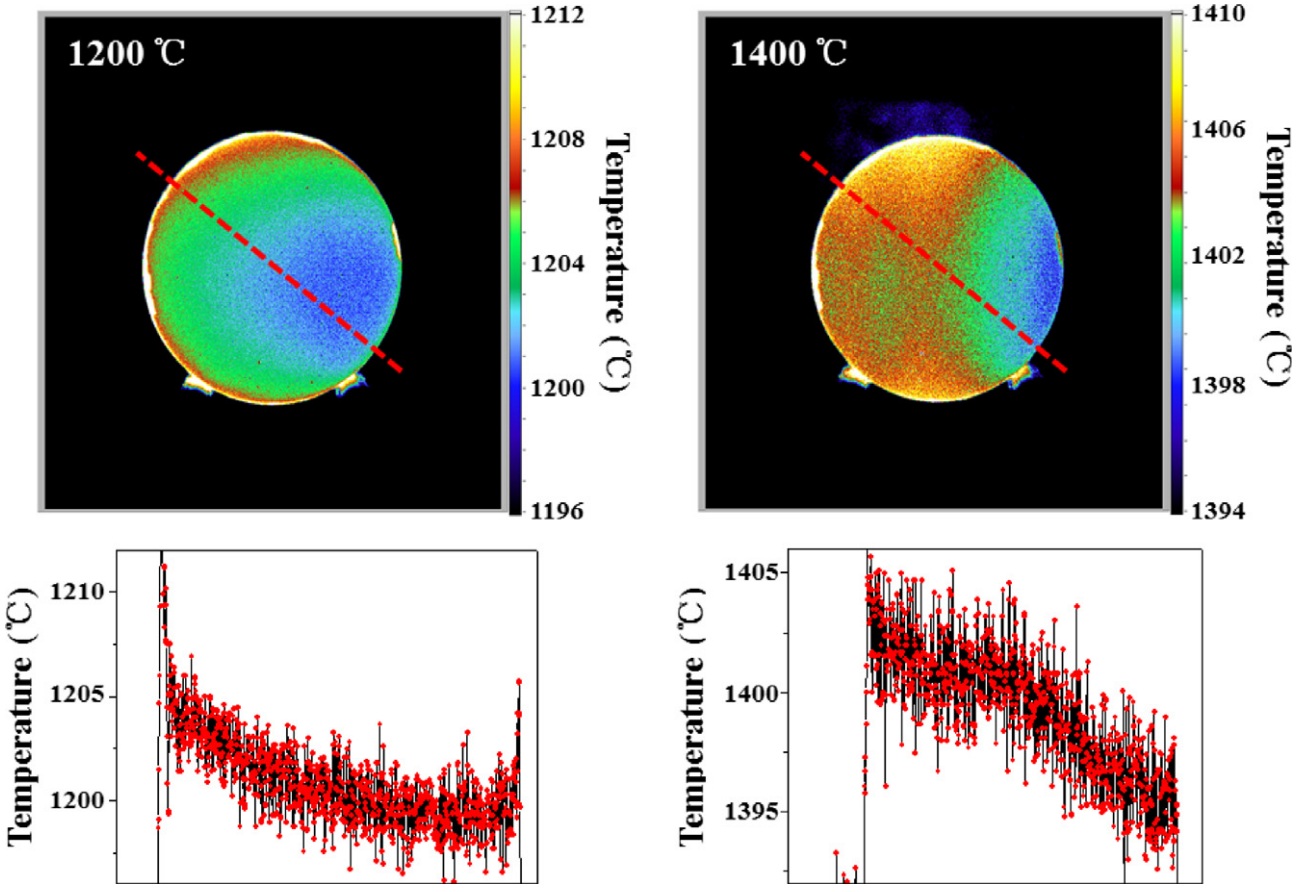

FIGURE 5 Temperature distribution and thermal profile for pure alumina at 1200°C and 1400°C [Color figure can be viewed at wileyonlinelibrary.com]

electromagnetic field has a weaker and weaker effect on the final densification.

3.2 | Evolution of thermal gradient on sample during microwave heating

The thermal gradient and profiles (along the dashed line) on the sample surfaces was evaluated during microwave heating with a heating rate of 25°C/min. for pure alumina and ZTA90 (Figure 5 and 6, respectively).

The thermal profile recorded at 1200°C for both compositions, clearly indicates heating from outside to inside of the sample. Here, the SiC susceptor plays the role of heating source. However, from 1300°C (for ZTA60 and ZTA90) or 1400°C (for pure alumina), an inversion of the gradient is observed. The temperature became higher in the center of the sample than at the edge. This indicates samples are coupling with electromagnetic field, due to the increasing dielectric loss of zirconia with temperature.[15] The results obtained for ZTA composites show that the thermal gradient inversion takes place at a lower temperature compared with pure alumina (about 100°C). This difference indicates that zirconia could act as an internal susceptor within composites and promote the heating homogeneity by combining the radiant heating from external SiC susceptor. This highlights the volumetric heating induced by microwave process, providing a more uniform microstructure.

3.3 | Optimal microwave sintering and characterization of dense ZTA composites

Based on the results presented in Figure 2, an experimental plan was established to define the ideal microwave sintering conditions (sintering temperature and holding time) to obtain dense materials. The best heating conditions as well as the final densities of microwave-sintered samples are shown in Table 2. According to the table, high densities have been obtained for ZTA composites under microwave sintering: 98.5% for ZTA97, 100% for ZTA90, and 98.1% for ZTA60. These dense specimens were then used for mechanical and microstructural characterizations in the next section.

Microwave-sintered samples microstructures were observed at first (Figure 7). The SEM micrographs of samples with differing amounts of zirconia exhibit zirconia (white) and alumina (dark) grains. These micrographs confirm the high densities of composites sintered by

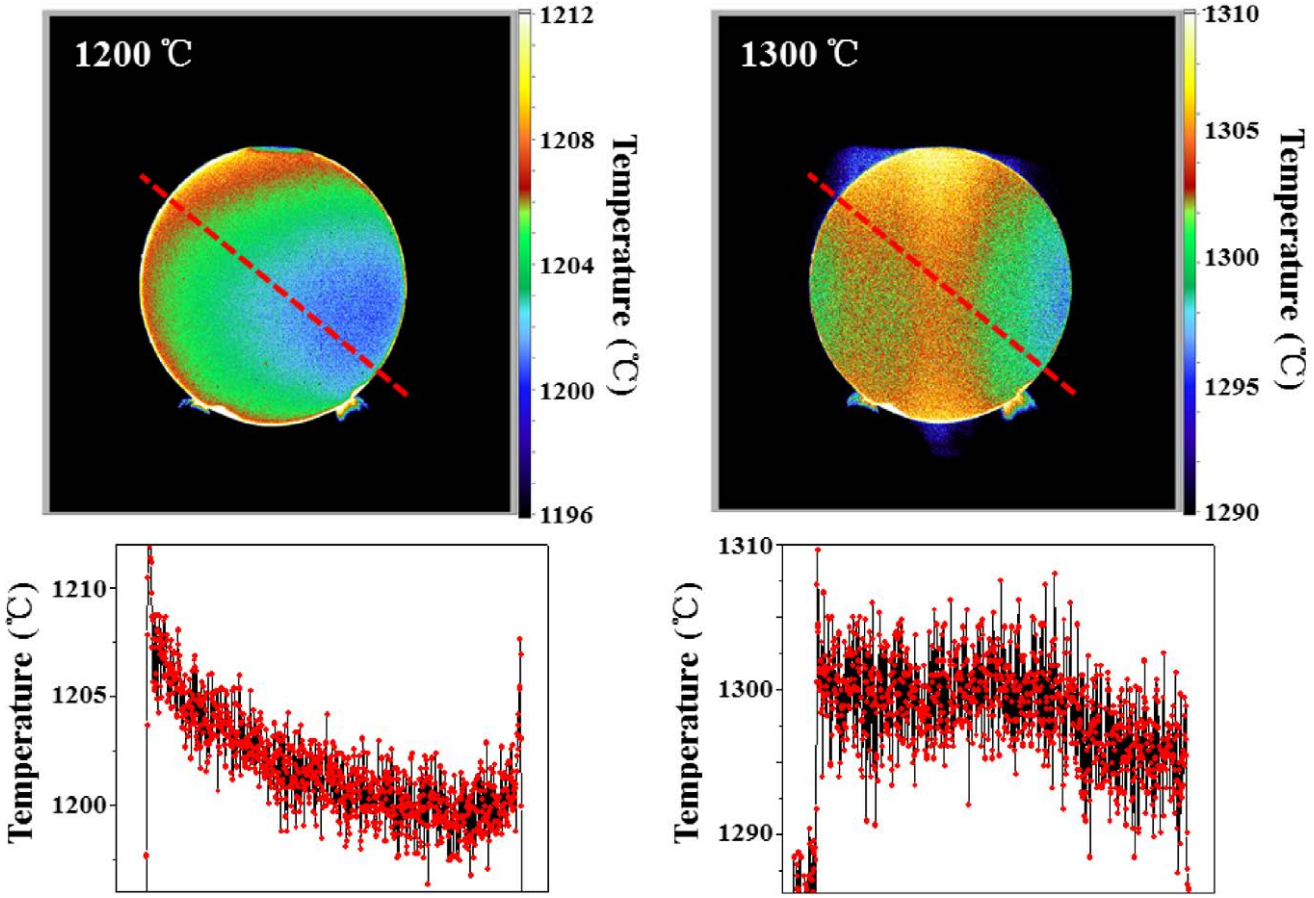

FIGURE 6 Temperature distribution and thermal profile for ZTA90 at 1200°C and 1300°C [Color figure can be viewed at wileyonlinelibrary.com]

microwave. Moreover, it can be mentioned that zirconia particles are uniformly dispersed in alumina matrix. In addition, the microstructure is homogeneous within samples, and no pore or abnormal growth of alumina grains can be found.

Figure 8 shows the evolution of alumina grain size as a function of zirconia content. When zirconia content increased, the grain size of alumina decreased significantly. When you look at the zirconia dispersion Figure 7, and combine that with the grain size (Figure 8), this fine microstructure is caused by the pinning effect of the zirconia phase around alumina grain that limits grain growth.[16] This phenomena improves the mechanical properties of composite. In summation, these results show the feasibility of applying microwave heating process to produce ceramic materials with high density and fine/homogeneous microstructure.

Vickers hardness of microwave-sintered dense sample is also presented in Figure 8. The maximum hardness value appears also in the case of ZTA90. Note, when the zirconia content increased from 10 to 40 vol%, the hardness declined even though the grain size continue to decrease. As a consequence, its hardness decreases. In fact, those results are the consequence of the balance of two effects: on one hand zirconia leads to a grain size decrease in the composite, that is favorable to the hardness (case of ZTA90), on the other hand zirconia hardness is lower than that of alumina, so it leads to a decrease in ZTA hardness (rule of mixture: case of ZTA60).

The evolution of fracture toughness vs zirconia content is plotted in Figure 9. Similarly the maximum fracture toughness was obtained in the case of ZTA90. This result achieved in both microwave sintering and conventional sintering[17] show the strengthening effect on the composite by zirconia.

TABLE 2 Experimental conditions for dense ZTA composites under microwave heating

	Heating rate (°C/min)	Sintering temperature (°C)	Dwell time (min)	Density (% T.D.)
ZTA97	25	1500	0	92.1
		1550	15	98.5
		1575	3	97.6
ZTA90		1500	0	94.1
		1550	15	100
		1575	5	99.6
ZTA60		1500	0	91.3
		1500	15	97.2
		1550	10	98.1

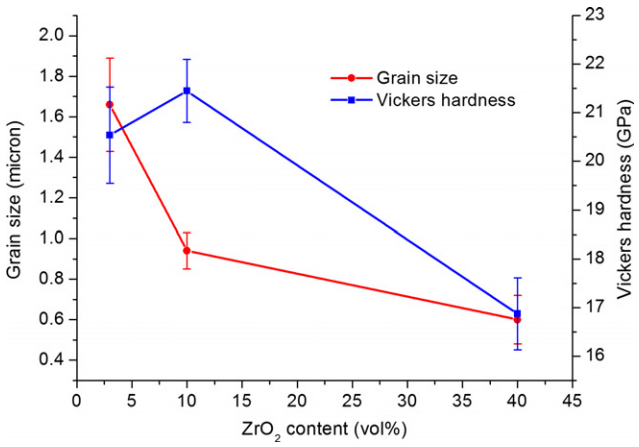

FIGURE 8 Variation in grain size and Vickers hardness on function of zirconia content [Color figure can be viewed at wileyonlinelibrary.com]

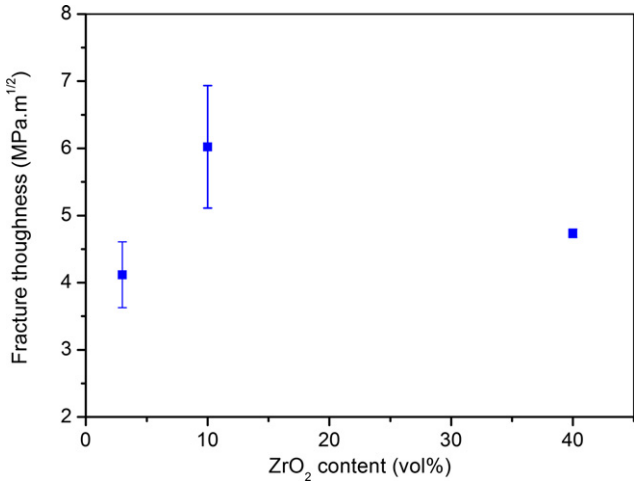

FIGURE 9 Variation in fracture toughness on function of zirconia content [Color figure can be viewed at wileyonlinelibrary.com]

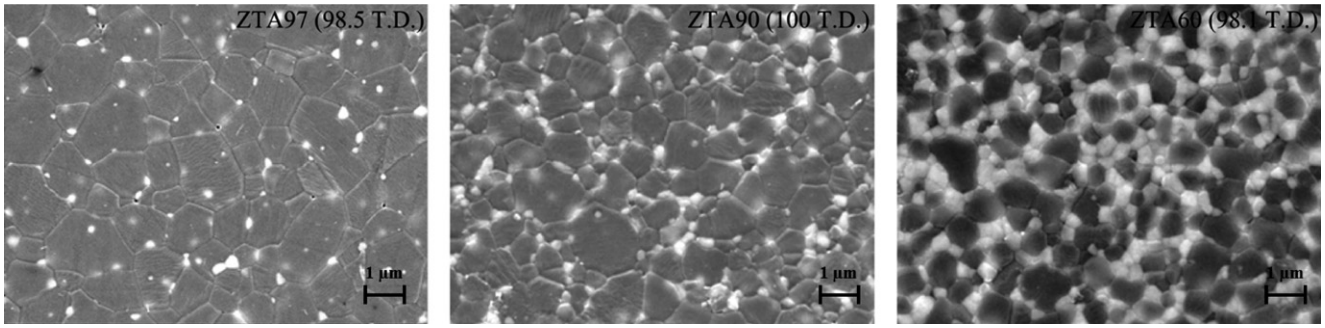

FIGURE 7 SEM micrographs of microwave-sintered dense ZTA composites

Finally, these mechanical properties of ZTA composites sintered in multimode microwave cavity agree with those reported in literature underlying single mode microwave heating process.[7] For example, the microwave-sintered ZTA90 (A10ZT in their paper) having a density of 99% of T.D. presented a Vickers Hardness of 19 GPa and a fracture toughness of 6 MPa $m^{1/2}$. For the same composition and by microwave sintering, we obtained in this study HV=21 GPa, and K_{1C}=5 ± 1 MPa $m^{1/2}$, respectively.

4 | CONCLUSION

A multimode-microwave hybrid furnace was used to sinter ZTA composites with various contents of zirconia. Rigorous comparison of densification behavior under identical thermal cycles between conventional and microwave sintering showed a expected delaying effect of zirconia on densification for both heating modes. This comparative dilatometric study demonstrated the beneficial effect of the electromagnetic field on densification of alumina and ZTA: under microwave, maximum densification rate occurs at lower temperature, leading to an increase in the sample density for any given temperature.

Analysis of thermal gradient on sample during microwave heating indicated that the presence of zirconia lowered the temperature when maximum temperature went from the surface to the interior. We attribute this phenomenon to the high microwave energy absorption via zirconia. Our work shows that hybrid microwave process combines both internal volumetric heating and external radiant heating, and hence favors the temperature distribution within the material, conducive to homogeneous microstructures. This latter effect is even more effective when zirconia is added into alumina, as it strongly couples with microwave.

After optimization the heating conditions, the feasibility to produce ZTA composite with high density through multimode microwave heating process was demonstrated. Zirconia phase inhibits the grain growth of alumina due to pinning effect. Moreover, a zirconia content of 10 vol% provides the ZTA composite the best mechanical properties.

The most significant aspect of our method is that multimode microwave sintering creates a finer homogeneous microstructure, with resulting high hardness and toughness as well as improved densification, and at lower cost.

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