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Microwave sintering of zirconia-toughened alumina (ZTA)-TiO_2-Cr_2O_3 ceramic composite: The effects on microstructure and properties

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ABSTRACT

This paper focuses on the development of a zirconia-toughened alumina ZTA-TiO_2-Cr_2O_3 ceramic composite by means of microwave sintering at 2.45 GHz within the range 1200 °C–1400 °C, with a dwell time of 5–20 min. It is aimed at attaining improved microstructure and properties at a lower sintering temperature and shorter soaking time, compared to using a conventional heating method. Consequently, the effects of sintering temperature and soaking time on densification, properties and microstructural behaviour of the composite, are investigated. XRD analysis reveals that the microwave-sintered samples possess a higher crystallinity at a higher sintering temperature. Microstructural analysis confirms the uniform distribution of particles and controlled grain growth; with the lowest AGI value being 1.28 grains/μm. The sample that is microwave-sintered at 1350 °C with 10 min of soaking time achieves a high density (95.74% of the theoretical density), elevated hardness (1803.4 HV), and excellent fracture toughness (9.61 MPa $m^{1/2}$), and intergranular cracks. This proves that the microwave sintering technique enhances densification, microstructural evolution and the properties of the ceramic composite at a lower temperature and shorter soaking time, compared to conventional heating. Overall, the improved mechanical properties of the microwave-sintered ceramics, compared to conventionally-sintered ceramics, are attributed to the enhanced densification and finer and more homogeneous microstructure that is achieved through the use of a microwave sintering method. The results reveal that microwave sintering is effective in improving the microstructure and density of materials, and will be useful for enhancing the mechanical properties of ZTA-TiO_2-Cr_2O_3 ceramic composites.

1. Introduction

Ceramic materials have been widely used in cutting industries due to their excellent mechanical properties. However, of more importance, is their ability to withstand higher temperatures than high-speed steel and carbide cutting tools [1,2]. Zirconia-toughened alumina (ZTA) has emerged as a new generation of toughened ceramics and is now one of the most widely used oxide ceramic structural composites [3]. ZTA, which is well-known for its excellent mechanical properties, such as high compressive strength (390 MPa) and toughness (4–13 MPa $m^{1/2}$) [4], is used for structural applications (i.e., cutting tools) and in many medical applications.

The author's previous works [5,6], successfully improved the properties of ZTA composites through the addition of TiO_2 and Cr_2O_3. However, the samples were subjected to conventional sintering; where they were exposed to a high sintering temperature for a long period. This resulted in undesirable grain coarsening and grain growth [7]. Consequently, the mechanical properties of ZTA were deemed critically dependent on its microstructure; which can

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be controlled by means of powder preparation and densification processes [8]. In order to produce a better product, it is extremely necessary to make improvements in the processing of the material.

Of the various sintering methods available, microwave sintering has attracted a great deal of interest in the processing of materials. Rapid heating can be achieved through microwave sintering, since the material is heated by energy conversion rather than energy transfer [9]. This contributes to volumetric heating [10]; thereby shortening the processing time and decreasing the densification temperature. It also accelerates the densification process and generates materials with more uniform and finer microstructures [11,12]. This leads to enhanced mechanical properties that will significantly improve the performance of the processed material [13]. Furthermore, from an economic perspective, microwave sintering is able to lower production costs thanks to energy savings through reduced sintering times [14].

This study is a continuation of the author's previous works [5,6], and is aimed at further enhancing the properties of ZTA-TiO_2-Cr_2O_3 by employing microwave sintering technology. A comparative study was also done, between the conventional sintering method and the microwave sintering method, to investigate the effects of each technique on the samples.

2. Experimental procedure

The raw materials used in this work were α-Al_2O_3 (99.8%, 1 μm, Martinswerk), yttria-stabilized zirconia (5.4 mol% Y_2O_3, 94.5%, 0.1–2.0 μm, Goodfellow), anatase-TiO_2 (>99.0%, 0.13 μm, Sigma Aldrich) and Cr_2O_3 (99.0%, <0.1 μm, Sigma Aldrich). The composition of each material was fixed at 3 wt.% (TiO_2) and 0.6 wt.% (Cr_2O_3), with the remaining mass containing Al_2O_3 and YSZ at a ratio of 4:1.

The raw materials were mixed according to their particular composition by ball milling them in acetone at a speed of 200 rpm for 1 h. The acetone provided fluidity for the powder during mixing and forming, and also served as a solvent for dissolving the additives to be uniformly incorporated into the powder, thereby yielding a homogeneous solution. The powder mixture was then placed in a dry oven at 80 °C for 24 h before being sieved. Green pellets were formed using a hydraulic hand press at a pressure of 300 N/mm^2.

The samples were subjected to microwave sintering in a microwave furnace (multimode cavity) at 2.45 GHz. The features of the microwave furnace that was used are shown in Table 1 below. A SiC susceptor was utilized to assist in the heating and sintering of the green samples. The microwave sintering temperature was set at various temperatures (1200 °C, 1250 °C, 1300 °C, 1350 °C and 1400 °C) with a holding time of 10 min. The optimum temperature achieved was subjected to different holding times (5 min, 10 min, 15 min and 20 min). The heating rate was set at 20 °C/min. After a definite isothermal holding time, the samples were slowly cooled to room temperature in the furnace at a cooling rate of 10–15 °C/min.

The phase composition and crystalline structure of the sintered samples were analysed by XRD (Bruker D8 Advance) with CuKα radiation operating at 40 kV and 30 mA. A scanning rate of 0.034°/s was maintained in the 2θ operating range of 20°–80°. The bulk density of the samples was calculated by means of Archimedes' principle. The microstructures of the samples were observed through a field emission scanning electron microscope (Supra 40 VP with EDX Genesis V5.10) and the grain sizes were measured according to the ASTM E-112 standard. The Vickers indentation technique was utilized to determine the Vickers hardness and fracture toughness of the sintered samples. Each sample was subjected to a 1-kgf load for 15s. The Vickers hardness of each sample was verified by taking the average of 5 different readings.

Table 1
Features of microwave furnace.

Power Supply	AC 220 ± 10 V 60 Hz
Rated Electric Power	3 kW
Output Power	0.2~1.45 kW variable
Microwave Frequency	2.45 GHz
Maximum Operating Temperature	1550 °C
Temperature Measurement	Infra-red pyrometer
Temperature Control	Automatic/manual, display profile
Effective Working Area	105 mm (L) × 105 mm (W) × 50 mm (H)

3. Results and discussion

3.1. XRD analysis

The XRD patterns of the ZTA-TiO_2-Cr_2O_3 ceramics prepared by microwave sintering are shown in Figs. 1 and 2. It can be seen that all the samples were composed of α-alumina (ICSD 98-004-0015) or corundum, ZrO_2 (ICSD 98-010-8646) and Al_2TiO_5 (ICSD 98-001-2288). The TiO_2 and Cr_2O_3 peaks were not detected in the XRD patterns, most likely due to the low content of the materials. Similar XRD patterns were detected for the samples at different sintering temperatures and dwell times. No formation of other new phases was observed throughout the experiments as the sintering temperature and dwell time increased.

It can be observed from Fig. 1 that the effect of varying the sintering temperature was an improvement in the peak intensity. The peak intensity of α-Al_2O_3 improved with an increase in the sintering temperature, thereby suggesting that the crystallinity of the ceramic was enhanced gradually. This result correlated well with the morphological results shown in Fig. 5, thus verifying that microwave sintering can promote grain growth in Al_2O_3. Overall, both the XRD results for different sintering temperatures and different dwell times showed no change in the number of peaks, indicating that all the crystal structures of the samples remained stable within the temperature range of 1200°C–1400 °C and dwell time of 5 minutes–20 min.

Very small scattered variations in the values of the c/a ratio, as shown in Fig. 3, indicated that the lattice distortion of Al_2O_3 was not significantly influenced by the sintering temperature. This might

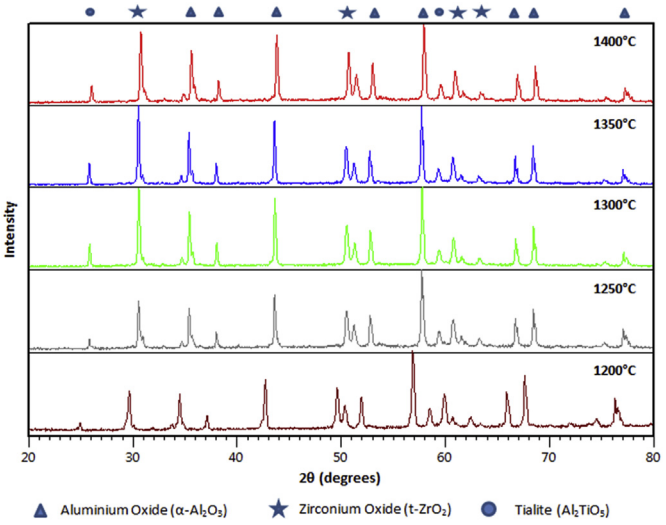

Fig. 1. XRD patterns of ZTA-TiO_2-Cr_2O_3 ceramic composite microwave sintered at different temperatures for 10 min.

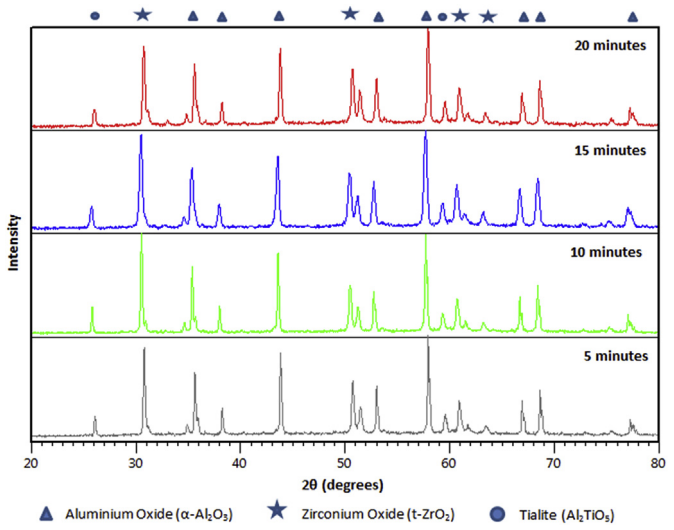

Fig. 2. XRD patterns of ZTA-TiO_2-Cr_2O_3 ceramic composite microwave sintered at 1350 °C with different dwell times.

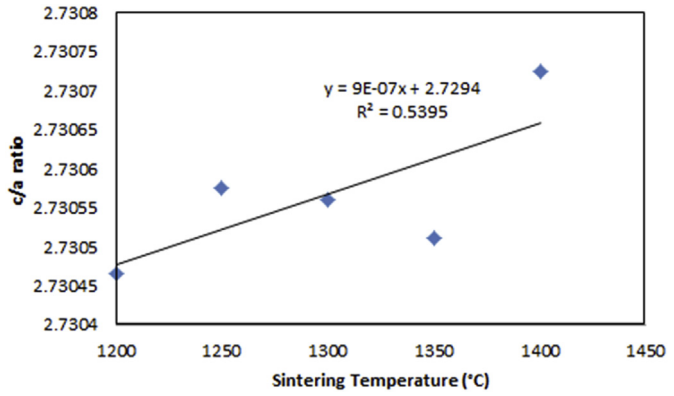

Fig. 3. Relation between c/a ratio and sintering temperature in microwave sintering.

have been due to the very low dielectric loss property of Al_2O_3 at a microwave radiation of 2.45 GHz [15]. However, the c/a ratio versus the sintering dwell time (Fig. 4) showed a certain trend, as demonstrated by the low slope of the c/a ratio trend line. The lattice distortion increased slowly but continuously with increasing dwell time. Such an increase in the lattice distortion was attributed to the

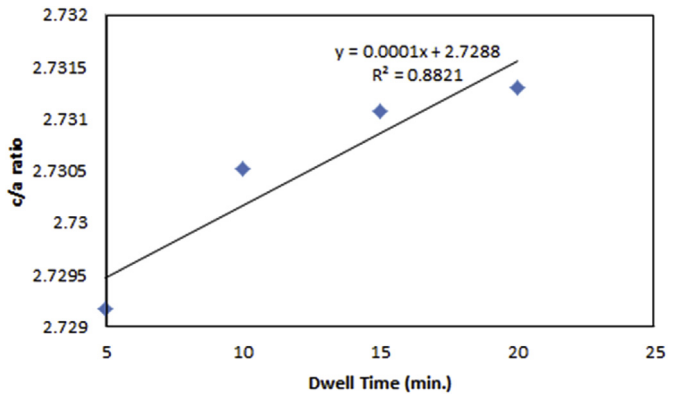

Fig. 4. Relation between c/a ratio and dwell time in microwave sintering.

constant dissolution of the Cr cations into the host alumina at the Al site.

3.2. Microstructural analysis

The microstructures of the microwave sintered samples at different temperatures are shown in Fig. 5. From the figure, remarkable densification and grain growth were observed when the samples were sintered to at least 1300 °C (Fig. 5(c)-(e)). The sample sintered at 1350 °C (Fig. 5(d)) showed very little porosity, had a homogeneous microstructure and a very compact grain arrangement. In contrast, the samples that had been microwave-sintered at 1200 °C (Fig. 5(a)) and 1250 °C (Fig. 5(b)) exhibited high porosity and an inhomogeneous grain distribution. This showed that temperatures below 1350 °C were insufficient for the densification and grain growth of this particular ceramic composite, thereby hampering the uniform heating of the samples. As a result of the poor dielectric property of Al_2O_3 and the moderate dielectric property of YSZ, as the main materials in the composite, the microwave absorption due to dielectric losses was negligible. Consequently, the primary heat transfer mechanism at low temperatures was blackbody radiation (from the susceptor), followed by microwave radiation at higher temperatures [16]. This caused a steep increase in the temperature of ZrO_2 after some time, while there was only a small increase in the temperature of Al_2O_3. Moreover, it was difficult to process ZrO_2 in a 2.45-GHz microwave furnace as the dielectric properties of this material changes rapidly with temperature and it has a very low thermal conductivity [17]. The uneven temperature distribution in the ZrO_2 particles impeded its densification, consequently initiating thermal runaway. However, ZrO_2 that has a higher content of dopant material (e.g., 10-YSZ) is known to be a better microwave absorber because of the higher ionic conductivity of ZrO_2 that is associated with the presence of higher oxygen vacancies in the lattice, which also contribute to the enhancement in the sintering process [15]. A study by Huang et al. [18] revealed that the use of nano-sized α-Al2O3 enhanced the dielectric properties of the samples and thus, achieved near theoretical density (>99%) when sintered at 1550 °C for 4 h without the addition of a sintering aid. It was also observed that pores started to form again at 1400 °C. This may have been due to the high temperature that caused the vaporization and condensation of Cr_2O_3 to CrO_3 [19] or the agglomeration of ZrO_2 particles [20].

Fig. 6 also shows that the sample sintered at 1350 °C for 10 min (Fig. 6(b)) had the least porosity and the most homogeneous microstructure compared to samples held for 5 min (Fig. 6(a)), 15 min (Fig. 6(c)) and 20 min (Fig. 6(d)) at the same sintering temperature (1350 °C). Very minor grain growth and less densification occurred in the sample held for 5 min due to the short soaking time. On the contrary, abnormal grain growth occurred in the samples with longer holding time (>10 min), causing an increment in the diffusion paths between the pores and the grain boundaries [21]. This resulted in the pores being entrapped within the large grains and hindering other grains from moving toward the pores. The formation of pores among the large grains following the longer holding time was also contributed by the agglomeration of ZrO_2 particles [20]. The ZrO_2 agglomeration, followed by the entrapment of the pores, led to the termination of the densification process. According to Figs. 5 and 6, the rapid densification, along with only minor grain growth that was achieved at 1350 °C and 10 min of soaking time, seemed to be an indication of the microwave effect [15].

The plot of the average grains intercept (AGI) vs. the sintering temperature is given in Fig. 7. The microwave-sintered sample obviously displayed a reduced AGI value (larger grain size) when exposed to a higher sintering temperature of up to 1350 °C. The AGI

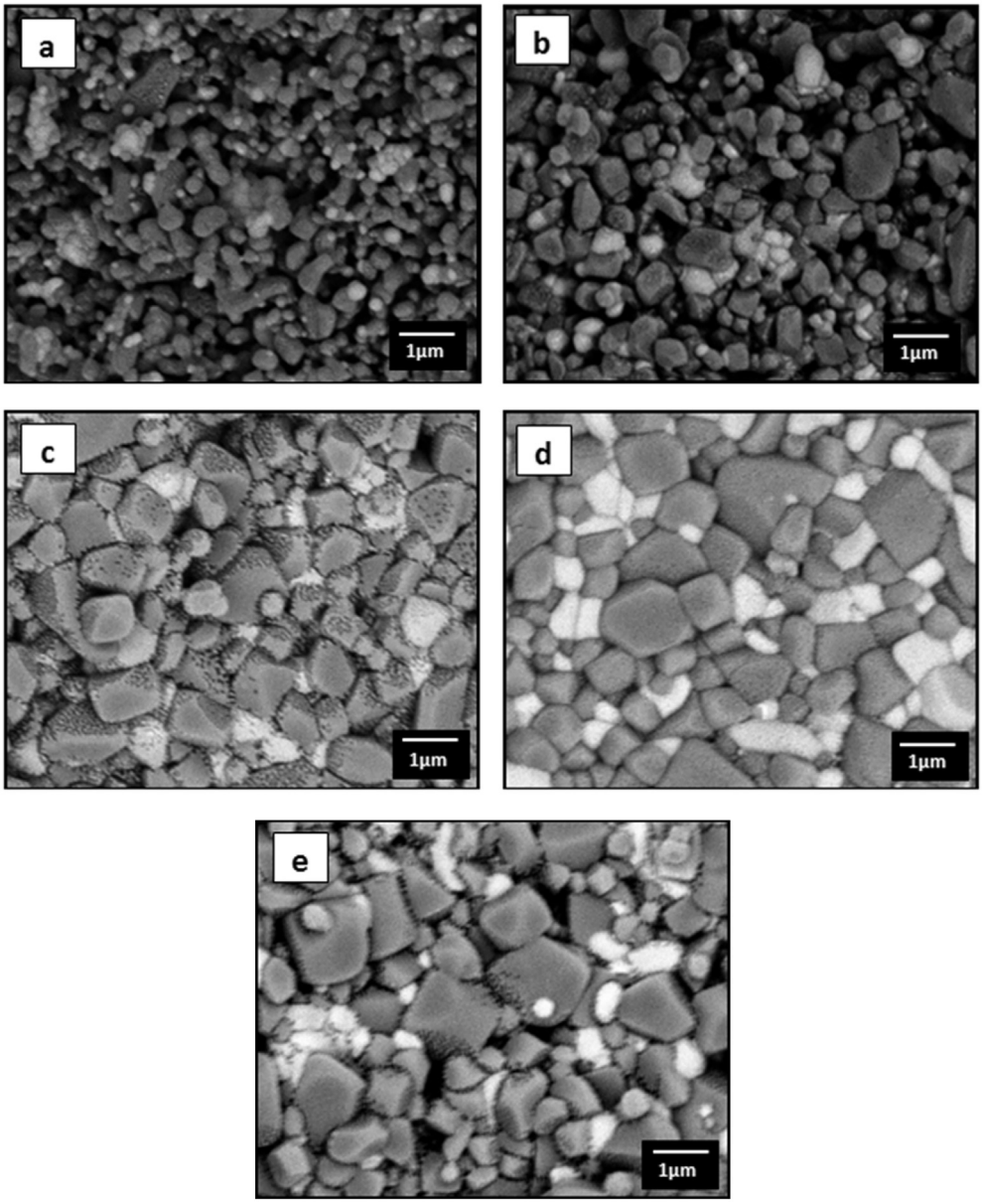

Fig. 5. Microstructures of samples microwave-sintered at (a) 1200 °C, (b) 1250 °C, (c) 1300 °C, (d) 1350 °C, and (e) 1400 °C, with 10 min of dwell time.

value was reduced from about 3.04 grains/μm at 1200 °C to 1.28 grains/μm at 1350 °C. These results indicated rapid grain growth accompanied by improved densification in the microwave sintering of the ceramic composite at considerably low temperatures. Xie et al. [22] also showed that a relatively large grain growth was observed below 1400 °C for high-purity submicron alumina processed by microwave sintering. Increasing the temperature beyond 1350 °C caused abnormal grain growth and pores entrapment, resulting in a slight increase in the AGI value.

Fig. 8 illustrates the influence of the holding time on grain growth at the sintering temperature of 1350 °C. It should be noted that after reaching the lowest AGI value with a holding time of 10 min, the grain sizes of the samples could not be increased further with a longer holding time, but instead the AGI value began to rise due to abnormal grain growth and pores entrapment. The rapid grain growth following the increase in the sintering temperature to 1350 °C and soaking time of up to 10 min may have been contributed by the increased coupling with the microwave fields and the grain boundary diffusion coefficients at the final temperature [23]. Janney et al. [24] reported that microwave sintering can greatly accelerate the growth kinetics of alumina and reduce the activation energy for grain growth compared to conventional sintering. The lower activation energy represents an acceleration of the diffusional process as a result of the microwave effect.

3.3. Density

Fig. 9 shows the density of ZTA-TiO_2-Cr_2O_3 ceramic composite microwave-sintered at different temperatures for 10 min. It was observed that the density increased with increasing sintering temperature up to 1350 °C, and started to drop beyond that particular temperature. This shows that grain growth is temperature dependent, where the primary mechanism of densification at lower temperatures is grain boundary sliding [25]. By keeping the heating temperature fixed at 1350 °C while varying the soaking time, the highest density was still obtained at 10 min of soaking

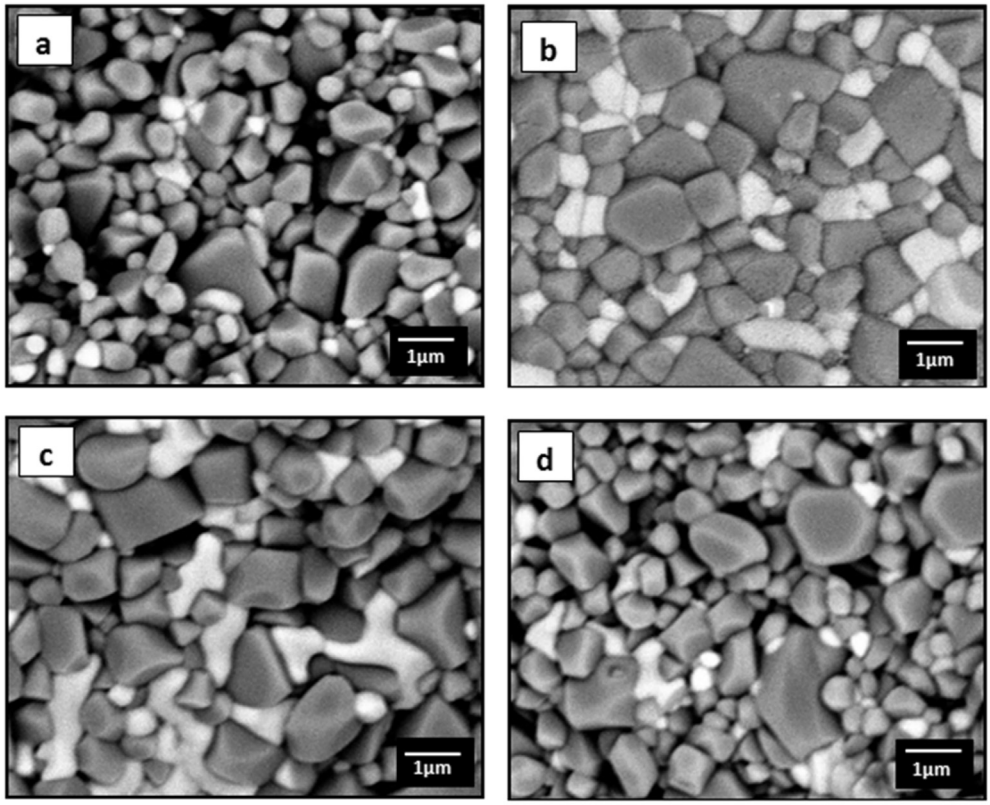

Fig. 6. Microstructures of samples sintered at 1350 °C for (a) 5 min, (b) 10 min, (c) 15 min, and (d) 20 min of dwell time.

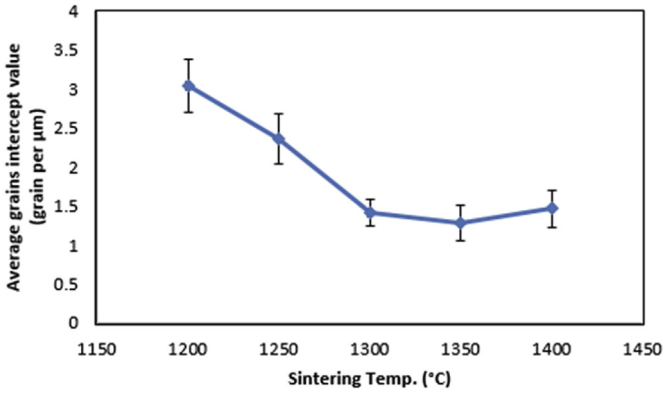

Fig. 7. Variation of AGI values with sintering temperature for 10 min of soaking time.

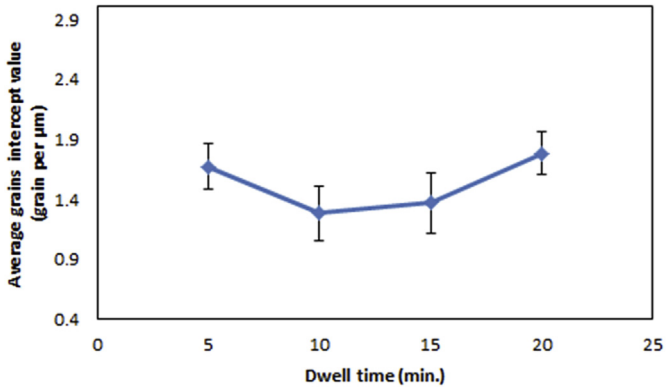

Fig. 8. Variation of AGI values with dwell time at sintering temperature of 1350 °C.

time, as indicated by Fig. 10. This result was parallel with the result obtained by Iddles et al. [26], where, after a certain holding time, any further increase in the sintering time resulted in a reduction in the density. The reduction in the ε_r (relative permittivity of the dielectric) value with increasing holding time was shown to affect the density of the material. In this work, the highest density achieved was about 4.18 g/cm^3, which was higher than the density of only 4.06 g/cm^3 obtained in the author's previous work [6], where conventional sintering was utilized. Volumetric heating by microwave sintering technology contributed to the high density. It was reported in the literature [15] that very small temperature gradients (<10 °C) were found between the centre and edge of the ceramic samples prior to microwave sintering. The high density of the materials can generate a high level of hardness due to the compact grain arrangement, as shown in Figs. 5(d) and 6(b).

The variations in the final density expressed as a percentage of the theoretical density with temperature and time are presented in Figs. 11 and 12. It can be seen that the microwave-sintered samples exhibited enhanced densification at a lower sintering temperature (1350 °C) and soaking time (10 min) compared to the conventionally-sintered samples (1600 °C for 1 h), as reported by Manshor et al. [6]. The microwave-sintered sample, for example, reached the highest value of 95.74% of the theoretical density at 1350 °C with a holding time of 10 min, whereas the conventionally-sintered sample reached the highest value of only 93.16% of the theoretical density at 1600 °C for 1 h of soaking time. The same density that was reached by the conventional sintering can be obtained by microwave sintering at a lower temperature (<1300 °C) with a shorter soaking time (10 min). This indicates that microwave sintering can accelerate densification, besides showing

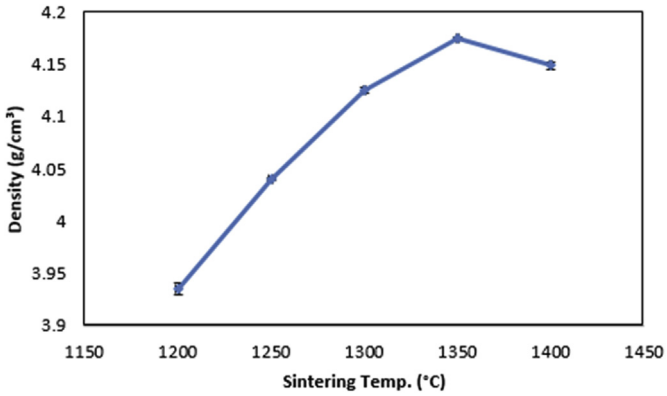

Fig. 9. Final density of microwave-sintered samples as a function of sintering temperature with 10 min of soaking time.

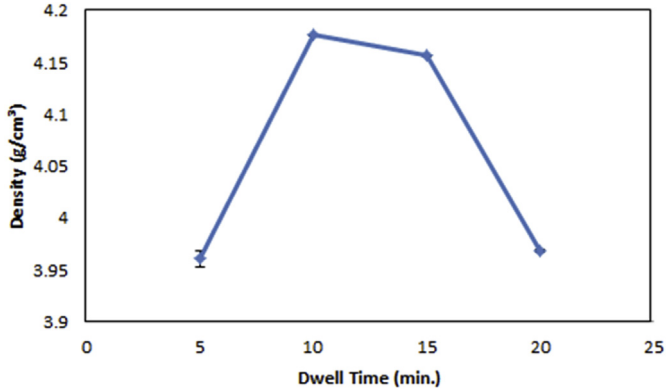

Fig. 10. Final density of microwave-sintered samples as a function of soaking time at a sintering temperature of 1350 °C.

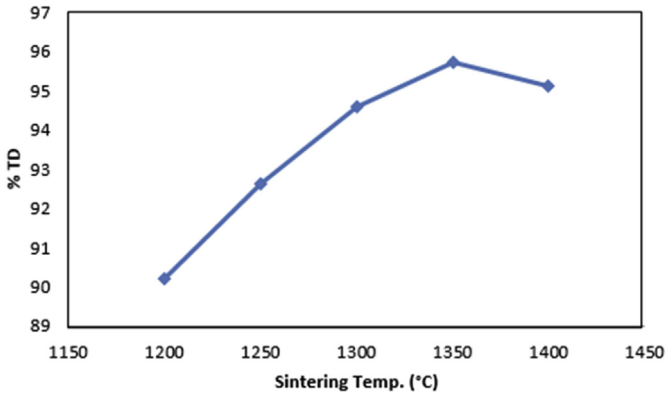

Fig. 11. Final density of microwave-sintered samples as a function of sintering temperature with holding time of 10 min.

a higher density than the conventional method, even though the sintering only occurs at 1300 °C for 10 min. Overall, the results verified that microwave sintering is a more favourable process for the promotion of densification than conventional sintering, provided that the sample is subjected to a suitable dwell time at a certain temperature.

On the other hand, after reaching the highest density value (≈96% of the theoretical density), no further increase in densification was observed. This result was parallel with an earlier work

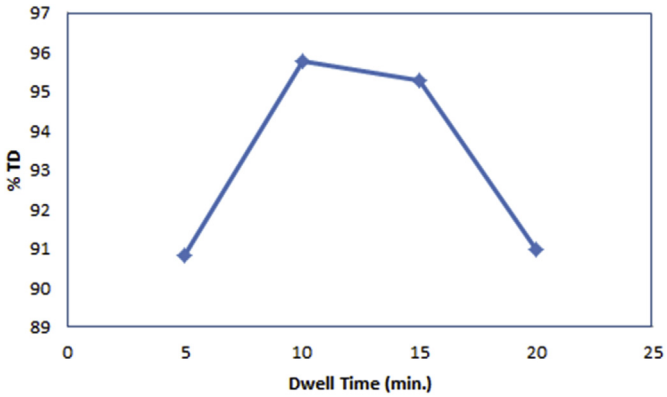

Fig. 12. Final density of microwave-sintered samples as a function of dwell time at a sintering temperature of 1350 °C.

by Xie et al. [20], where it was suggested that further densification could not be realized due to the agglomeration of ZrO_2 particles. It was also explained that the crystallites were initially sintered due to the structure of the polycrystals, until the sintering of the ZrO_2 agglomerates became complicated in the microwave radiation. A higher density value can only be attained if the agglomerates are eliminated.

3.4. Hardness and fracture toughness

The Vickers hardness of the microwave-sintered samples are presented in Fig. 13 as a function of the sintering temperature, and in Fig. 14 as a function of the soaking time (at a sintering temperature of 1350 °C). The hardness data trend was consistent with the density data, as shown in Figs. 9–12. The maximum hardness of 1803.4 HV was obtained for the sample sintered at 1350 °C for a holding time of 10 min. It has been stated that the ceramic gets harder when the grain size is smaller [27]. However, this work revealed that grains that were too fine due to incomplete sintering led to a reduction in the hardness value. High porosity as a result of incomplete sintering and pore coalescence [28] also contribute to extremely low hardness values.

The fracture toughness of the microwave-sintered samples that were subjected to different sintering temperatures and soaking times is shown in Figs. 15 and 16, respectively. It can be seen that the fracture toughness of the samples increased at first with temperature and time. The maximum fracture toughness of 9.61 MPa $m^{½}$ was obtained at 1350 °C for a holding time of 10 min,

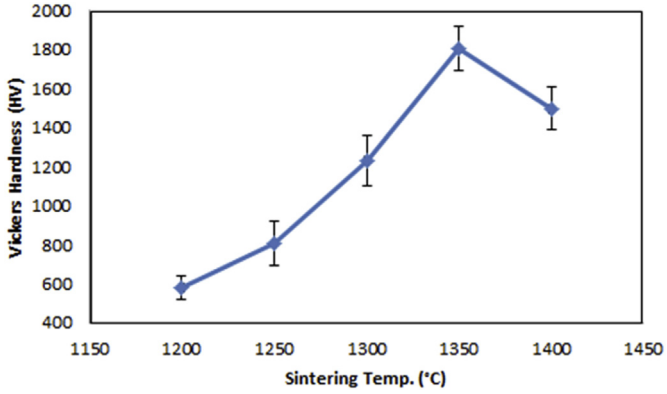

Fig. 13. Vickers hardness value of microwave-sintered samples as a function of sintering temperature for a holding time of 10 min.

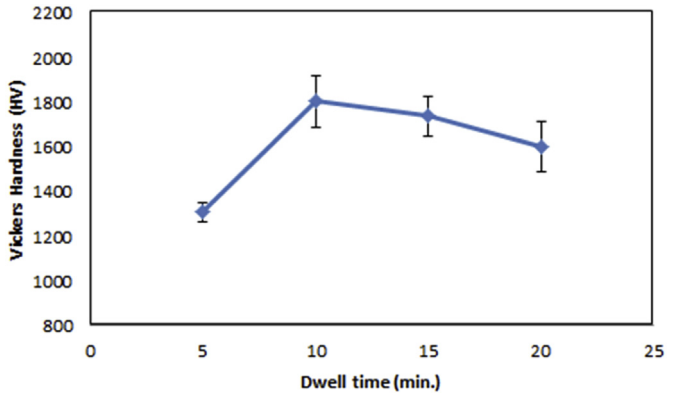

Fig. 14. Vickers hardness value of microwave-sintered samples as a function of dwell time sintered at 1350 °C.

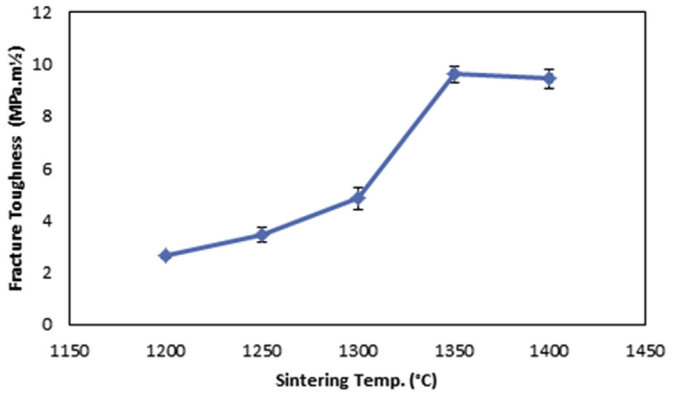

Fig. 15. Fracture toughness of microwave-sintered samples as a function of sintering temperature for a holding time of 10 min.

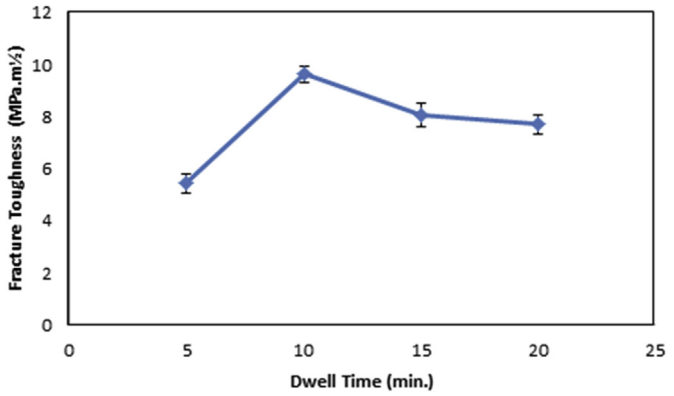

Fig. 16. Fracture toughness of microwave-sintered samples as a function of dwell time sintered at 1350 °C.

which was in accordance with the microstructure of the sample revealed by the SEM image.

The complete sintering process improved the density, microstructure and mechanical properties of the sample. The maximum fracture toughness of the sample that had been microwave-sintered at 1350 °C for 10 min was attributed to its homogeneous, fine-grained and dense microstructure. It was found that the rise in the sintering temperature and soaking time beyond 1350 °C and 10 min in the microwave sintering process resulted in a reduction in the fracture toughness value, probably due to a significant increase in the number of pores and pronounced abnormal grain growth.

Table 2 shows the properties of the ZTA-TiO_2-Cr_2O_3 ceramic composite prepared by microwave sintering and conventional sintering. The mechanical properties of the microwave-sintered ceramic composite (1350 °C, 10 min) were further enhanced compared to those of the conventionally-sintered ceramic composite reported by Manshor et al. [6]. For example, the Vickers hardness and fracture toughness of the microwave-sintered ceramics were 7.28% and 44.9% higher, respectively than those of the conventionally-sintered ceramics. The hardness of the ceramics was known to increase correspondingly with its density. Compared to the final density of the conventionally-sintered sample [6], the microwave-sintered sample (1350 °C, 10 min) was 2.77% denser. Additionally, the empirical relation between the hardness and the grain size in the ceramics suggests that a smaller grain size will produce harder ceramics. The final grain size obtained by the microwave-sintered ceramic composite at 1350 °C for 10 min was much finer than that of the conventionally-sintered ceramics. For that reason, the Vickers hardness value of the microwave-sintered ceramics was definitely higher than that of the conventionally-sintered ceramics. The higher fracture toughness of the ZTA-TiO_2-Cr_2O_3 ceramic composite obtained in this work was also mainly determined by the improved relative density of the ceramics compared to the conventionally-sintered ceramics. The denser microwave-sintered ceramics yielded a higher fracture toughness value. These results were not parallel with the results obtained by Goldstein et al. [29], which showed similar mechanical properties between the YSZ samples sintered by the microwave and conventional processes. This might have been due to the sintering of a single phase without the addition of other material additives.

3.5. Crack analysis

Fig. 17 shows the surface morphologies of the ZTA-TiO_2-Cr_2O_3 ceramic composites prior to the Vickers hardness test. There were distinct differences between the microstructures of the two samples. The surface morphologies revealed that many pores were observed in the sample that had been microwave-sintered at 1200 °C (Fig. 14 (a)). However, the sample that had been microwave-sintered at 1350 °C (Fig. 14 (b)) showed only a minimum number of pores. The fractographs of these two samples were also obviously different. Apparently, grain growth following an increase in the sintering temperature influenced the fracture mode of the samples. Extremely small grain sizes were obtained as the heating temperature was insufficient to activate the grain growth

Table 2
Mechanical properties of ZTA-TiO_2-Cr_2O_3 ceramic composite prepared by microwave sintering and conventional sintering.

Sintering process	Sintering Temperature (°C)	Soaking Time (min)	Vickers Hardness (HV)	Fracture Toughness (MPa.m^{½})
Conventional (Manshor et al. [6])	1600	60	1680.97	7.15
Microwave	1200	10	581.16	2.63
Microwave	1250	10	806.22	3.44
Microwave	1300	10	1232.4	4.85
Microwave	1350	5	1305.22	5.41
		10	1803.4	9.61
		15	1737.46	8.05
		20	1599.2	7.68
Microwave	1400	10	1496.2	9.44

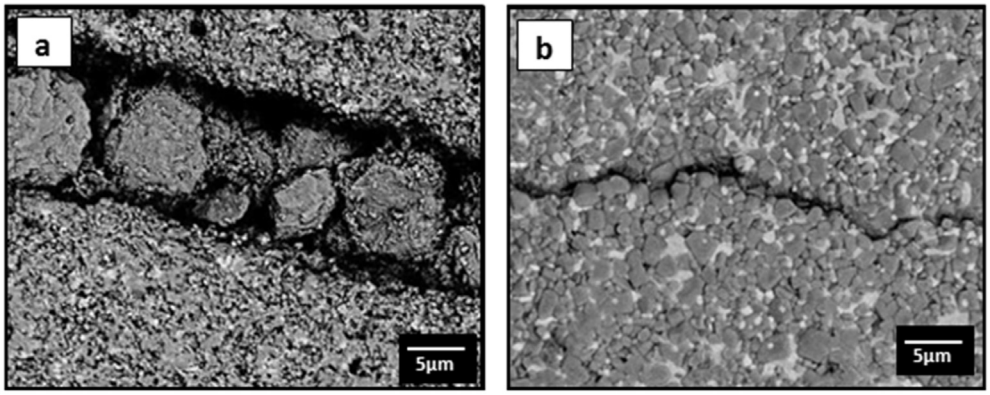

Fig. 17. SEM micrographs of the surface of two ZTA-TiO2-Cr2O3 ceramic composites microwave-sintered at (a) 1200 °C and (b) 1350 °C, for a soaking time of 10 min.

process, thereby contributing to the brittleness of the material. On the other hand, the sintering temperature of 1350 °C and soaking time of 10 min produced adequate grain sizes for crack deflection, thus resulting in an intergranular fracture mode. The suitable micro-roughness of the surface due to grain growth forced the crack to propagate in a crooked crack path, instead of in a straight crack path, so as to bypass the rigid grains, consequently leading to a reduction in the crack driving force.

4. Conclusion

ZTA-TiO_2-Cr_2O_3 ceramic composite was successfully fabricated by the microwave sintering method. However, it should be noted that the success of this method depends on the sintering conditions (sintering temperature and soaking time). A suitable balance between temperature and time will allow sufficient time for minimal grain growth, as long as a highly dense and homogeneous microstructure is obtained. For microwave-sintered ZTA-3 wt% TiO_2–0.6 wt% Cr_2O_3, the optimum mechanical properties were obtained at a temperature of 1350 °C and soaking time of 10 min, where the density, Vickers hardness and fracture toughness were 95.74% of the theoretical density, 1803.4 HV and 9.61 MPa $m^{½}$, respectively. Compared to the conventional sintering technique performed by Manshor et al. [6], the material produced by the microwave sintering technique demonstrated better mechanical properties. The properties were significantly higher than those of the conventionally-sintered sample by as much as 2.77% (density), 7.28% (Vickers hardness) and 44.9% (fracture toughness). This could be attributed to the volumetric heating, which contributed to the lower sintering temperature and shorter sintering time. Thus, this resulted in the fabrication of a material with a finer grain size, improved densification and excellent mechanical properties.

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