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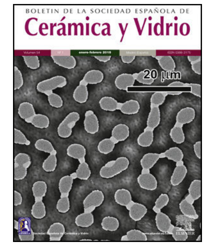

Microwave versus conventional sintering: Microstructure and mechanical properties of Al_2O_3–SiC ceramic composites

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ABSTRACT

In this report, Al_2O_3–SiC ceramic composites were produced at 1500 °C by conventional and microwave sintering. For preparing samples, Al_2O_3 with the second phase x wt.% SiC (x = 5, 10, 15, 20) were milled for 180 min. The milled powders were compacted in uniaxial press at 60 MPa for 30 s and sintered by both the conventional and microwave sintering methods. After sintering, densification, grain size, hardness, fracture toughness, phase variation and microstructure of the samples were examined, and comparisons were made for both the sintering methods. The experimental results revealed that there was an increase in density in the microwave sintering method when compared to conventional sintering. However, it was found that the density decreased in both the conventional and microwave sintering methods when there was an increase in SiC content. The highest relative density of 99.7% was obtained in 5 wt. % SiC composite produced by microwave sintering. With regard to hardness and fracture toughness, in both the microwave and conventional sintering methods, though they increased initially and they decreased when there was an increase in SiC content. The maximum hardness and fracture toughness of 24.6 GPa and 5.7 MPa $m^{1/2}$, respectively, was obtained in 10 wt. % SiC composite sintered by microwave sintering. In both the sintering processes, X-ray diffraction pattern shows the formation of a SiO_2 phase in all four compositions along with Al_2O_3 and SiC phases in conventional sintering, but in microwave sintering only negligible amount of SiO_2 phase formed in 15 and 20 wt.% SiC composites. The crystalline size decreases in microwave sintering than conventional sintering due to shorter sintering time. Uniform agglomeration and fine grains in the range of 2–3.6 μm were formed in microwave sintering, whereas grain size decreases with an increase in the SiC content due to grain boundary pinning due to the intergranular SiC particle.

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Microondas versus sinterización convencional: microestructura y propiedades mecánicas de compuestos cerámicos Al_2O_3–SiC

Palabras clave:
Al_2O_3–SiC
Compuesto de cerámica
Microondas
Sinterización

RESUMEN

Se estudian materiales compuestos de Al_2O_3–SiC procesados por sinterización convencional a 1500 °C y por microondas. Las mezclas de Al_2O_3 con la segunda fase *x*% en peso de SiC (*x* = 5, 10, 15, 20) se prepararon por molienda en molino de bolas durante 180 minutos, secado y prensado uniaxial a 60 MPa durante 30. En ambos procesos de sinterización, el patrón de difracción de rayos X muestra la formación de fases de SiO_2 junto con las fases originales, Al_2O_3 y SiC, siendo mayor su proporción en los materiales preparados por sinterización convencional y mayores proporciones de SiC. Los materiales sinterizados se caracterizaron en términos de densidad, fases cristalinas, microestructura, tamaño grano, dureza y tenacidad a la fractura Vickers. Se realiza una comparación entre los materiales preparados utilizando ambos métodos de sinterización. La sinterización de microondas proporciono materiales de mayores densidad, dureza y tenacidad que la sinterización convencional. El máximo de densidad se obtuvo para 5% en peso de SiC y los valores disminuyeron para mayores proporciones de segunda fase. Los máximos de dureza y tenacidad a la fractura se obtuvieron para 10% en peso de SiC.

Introduction

In many industrial fields, Al_2O_3 ceramics are extensively used. However, mechanical properties of Al_2O_3 are in downturn because of the bizarre grain growth during sintering. Because of its brittle nature, Al_2O_3 applications can be narrowed [1]. There are numerous methods to escalate the mechanical properties of Al_2O_3. One of the methods is adding SiC as a second-phase particle. Significant changes in mechanical properties are noticed by the presence of SiC as a second-phase particle [2]. When compared with monolithic ceramics, ceramic composites show significant improvement in mechanical properties. Structural applications of Al_2O_3 require superior mechanical properties that have been obtained by sintering it to full dense structure. In recent times, requirement of Al_2O_3 ceramic composites expanded and a variety of approaches have been introduced in sintering process to produce full dense structure. Mostly, these composites are fabricated by pressureless sintering [3,4], hot press sintering [5] and hot isostatic pressing sintering [6] to produce full dense composites. In pressureless sintering, parameters such as low heating rate, higher sintering temperature and long holding time only help to produce the full density ceramic composites. But the long holding time and high temperature lead to abnormal grain growth, resulting in the decline of properties. Moreover, pressureless sintering requires high energy and time to fabricate the ceramic composite. The density and mechanical properties of ceramics are found to be high in hot press and hot isostatic pressing sintering than in pressureless sintering owing to the pressure applied in the former processes. But both the hot press and hot isostatic pressing sintering processes are not suitable for producing complex shapes and are not cost-effective. Therefore, they are not desired for mass production.

In order to reduce above limitations, microwave sintering is used. Globally, the microwave sintering process has pulled the interest of researchers in recent decades [7–9]. Conductive and convective heating phenomena by radiant heating element involved in conventional sintering, whereas volumetric heating in microwave sintering. Compared to conventional sintering, microwave sintering is regarded as a powerful technique because with reduced energy consumption it can contribute improved properties with finer microstructure [10,11]. The numerous studies comparing conventional and microwave sintering show reduced sintering temperature in microwave sintering because microwave increases the atom kinetic energy, accelerates the grain-boundary diffusion and increases the densification rate [12,13]. High hardness and positive mechanical properties have been produced successfully for Al_2O_3-based ceramics by microwave heating [14–19].

Monolithic Al_2O_3 with 99.7% density and grain size of ~2.5 μm were produced by microwave sintering with shorter sintering time. Grain growth increases associated with fast densification were observed in microwave sintering [15]. Brosnan et al. found that microwave-sintered monolithic Al_2O_3 reached almost full density at 1400 °C, whereas only 52% density was obtained in conventional sintering. During microwave sintering densification starts at 1100 °C, but in conventional method it starts at 1300 °C. Approximately 250 °C shift for densification process was present between both the methods. Full density was achieved in both the method at 1600 °C but holding time for conventional sintering was more than microwave sintering. In both the methods, almost same grain size was produced at 1600 °C [18]. More studies were carried out in microwave-sintered monolithic Al_2O_3. In order to enhance the mechanical properties, a secondary phase element was added with Al_2O_3. It is well known that Al_2O_3/SiC ceramic composites show better mechanical properties than

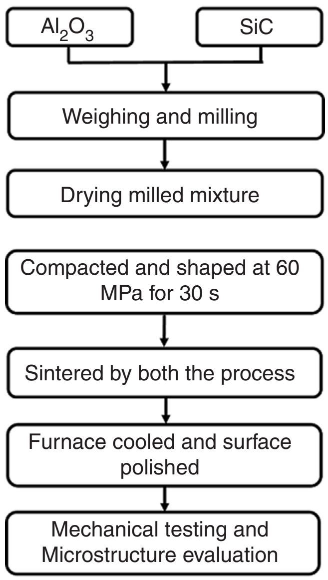

Figure 1 – Process flow chart for sample preparation.

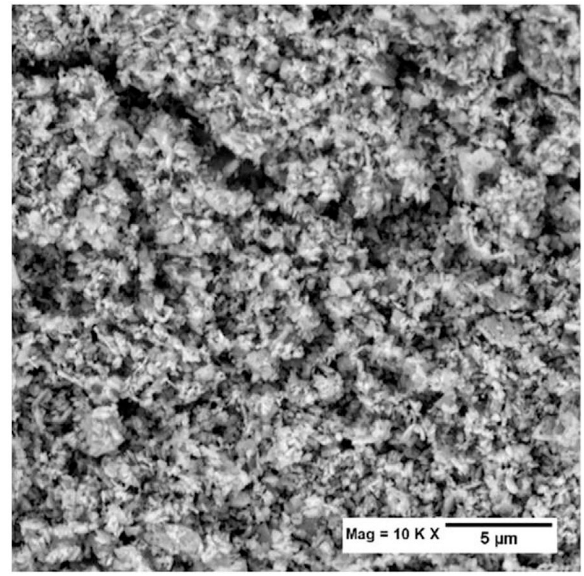

Figure 2 – The SEM image of ball-milled powder before compaction.

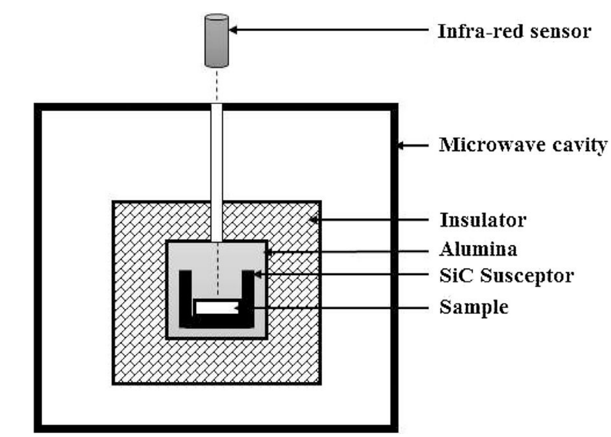

Figure 3 – Arrangement of the susceptor and sample for microwave sintering.

monolithic Al_2O_3. A number of researchers have found that the presence of relatively small amount of SiC enhances the mechanical properties of alumina, such as hardness [20,21] and fracture toughness [22] at room temperature. Al_2O_3/SiC ceramic composites with high mechanical properties are commonly produced by hot press sintering. However, hot pressure sintering and other conventional processes need high sintering temperature and time to enhance the properties.

In order to overcome the said limitations in the present study, we attempted to fabricate Al_2O_3/SiC ceramic composites by microwave sintering and expected to obtain enhanced mechanical properties. Effect of microwave sintering on relative density, average grain size, hardness, fracture toughness and microstructures were also investigated. In addition to this, comparison between the microwave and conventional sintering was also conducted.

Experimental

Commercially available α-Al_2O_3 (sigma Aldrich) and β-SiC (sigma Aldrich) powders were used in this study in which alumina powder has the purity of 99.5%, average grain size of 3 μm; SiC has the purity of 99% and average grain size of 1 μm. Fig. 1 shows the various steps involved in the preparation of the Al_2O_3/SiC ceramic samples. Al_2O_3/x wt.% SiC powders (x = 5, 10, 15, 20) were mixed and ball milled with isopropyl alcohol using tungsten carbide (WC) ball at 300 rpm for 180 min. The mixture was dried at 80 °C and sieved. Fig. 2 shows the SEM image of milled powder before compaction. The homogeneous mixtures were compacted into cylindrical pellets of 15 mm diameter with 5 mm thickness by cold uniaxial press at 60 MPa for 30 s. Four such pellets were made from each of the four compositions. One set of compacted pellets was sintered in an electric resistance heating furnace ($MoSi_2$ Heating Elements) at 1500 °C with a holding time of 300 min and a heating rate of 10 °C per minute. Another set of compacted pellets was also sintered at 1500 °C in a microwave furnace at 2.45 GHz (magnetron heating element) using susceptor materials as auxiliary heating elements. Input power ranging from 0.9 to 2.4 kW and holding time of 15 min were used. The temperature in microwave furnace was measured by a non-contact type infrared sensor and controlled by a Eurotherm (Model 2416) microprocessor-based PID controller with a digital indicator. Fig. 3 represents the arrangement of the susceptor and sample for microwave sintering. In both the processes, pellets were cooled in the furnace itself. After sintering and cooling, the surface of the pellets was polished in lapping machine using diamond paste.

Density of the pellets was measured by using Archimedes' principle by immersing in distilled water based on ASTM B311 standard. For determining the average grain size, the intercept method was used. Hardness H_v of the samples was measured by Vickers indentation method based on the ASTM C1327 standard with indentation load of 5 kg for 30 s. Fracture toughness was calculated using indentation method which was

Table 1 – Relative density and matrix grain size with SiC content for both the sintering methods.

Samples	Relative densities (%)		Average matrix grain size (μm)	
	Conventional	Microwave	Conventional	Microwave
Al_2O_3/5 wt.% SiC	98.2	99.7	~4.7	~3.6
Al_2O_3/10 wt.% SiC	97.9	99.6	~3.5	~3.2
Al_2O_3/15 wt.% SiC	96.4	99.2	~2.3	~2.5
Al_2O_3/20 wt.% SiC	95.8	98.7	~1.8	~2.0

mostly used by the researches because of its advantages over the conventional methods like the experimental procedure is straightforward, involving minimal specimen preparation and small amount of material. But, care should be taken while calculating the crack length and diagonal length of the indentation. In this paper, fracture toughness K_{IC} were calculated by the Vickers indentation method, given by Evan's equation

$$K_{IC} = 0.203 H_V a^{1/2} \left(\frac{a}{c}\right)^{-3/2}$$

where $2a$ and $2c$ are the diagonal length of the indentation and total crack length, respectively [20]. The various phases after the sintering process were identified by using X-ray diffractogram (XRD-SMART lab, JAPAN) using Cu K beta radiation with 45 kV and 30 mA as working parameters. The scan range, stepping angle and scan speed were 20° to 80°, 0.02° and 4° per minute, respectively. The crystallite size was evaluated through Scherrer method by applying the following equation [23]:

$$d = \frac{0.9\lambda}{b \cos\theta}$$

where b, θ, λ and d are the full width of the peak at half intensity, position of peak in the pattern, the wave length of X-ray and crystalline size. In order to study the microstructure and grain properties, the image obtained from field emission scanning electron microscope (FESEM-SUPRA 55, Carl Zeiss, Germany) was used.

Results and discussion

Relative density and grain size

Table 1 shows the basic characteristics such as relative density and average matrix grain size of both the conventional and microwave-sintered samples. Relative density was calculated based on the theoretical densities 3.99 and 3.21 g/cm^3 of Al_2O_3 and SiC, respectively. 5 wt.% SiC ceramic had the highest relative density of 98.2% in conventional and 99.7% in microwave sintering. In both the conventional and microwave sintering methods, the relative density decreases with increase in the SiC content. This is mainly due to the inclusion of SiC particles that block the grain boundary movement and inhabited the densification of Al_2O_3. In all the cases, microwave sintering shows higher density than conventional sintering as microwaves have the potential to produce higher densities at lower temperature in the ceramic system. Preceding reports also show that microwave sintering has been perceived as a promising method to enhance the densification of ceramics [13,24–26]. In order to achieve high density in conventional sintering, higher sintering temperature and higher pressure are required.

Table 1 shows the variations of matrix grain size with SiC content for both conventional and microwave sintering. Significant refinement in grain structure was observed with the increase in SiC content for both the sintering methods. The larger grain size was observed in 5 and 10 wt.% SiC content, because less intergranular SiC particle produces less grain boundary pinning. As the amount of SiC content increases (15 and 20 wt.% SiC content), the number of intergranular SiC particles also increases which reduces the grain growth due to increase in the effective grain boundary pinning [27]. In microwave sintering, for all the samples, suppressing grain growth was observed due to the intergranular SiC particle which hinders the forward diffusion of inter grain ions between alumina particles. But uniform grain formation takes place in microwave sintering than conventional sintering [24,25].

Hardness and fracture toughness

The variations of Vickers hardness versus SiC content are shown in Fig. 4. It shows that in both conventional and microwave sintering, Vickers hardness increases with increasing content of SiC up to 10 wt.% due to smaller grain size of the composite and the presence of hard secondary-phase SiC. Further increase in SiC content decreases the hardness due to low relative density which may have a negative effect on the hardness [21]. Conventional sintered specimen shows lower Vickers hardness than microwave sintering because of the presence of residual internal stresses produced by the thermal expansion mismatch between Al_2O_3 and SiC [26]. Vickers hardness decreases with 15 and 20 wt.% SiC because of the agglomeration of SiC in the ceramic composite which also

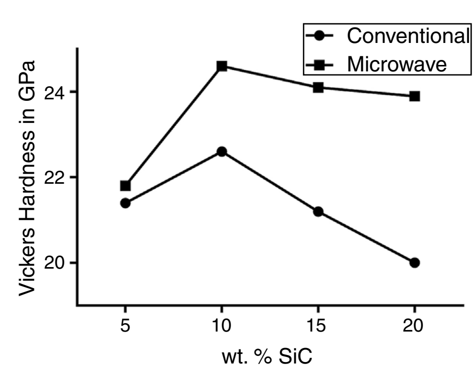

Figure 4 – Vickers hardness for the Al_2O_3/x wt.% SiC samples by both sintering methods.

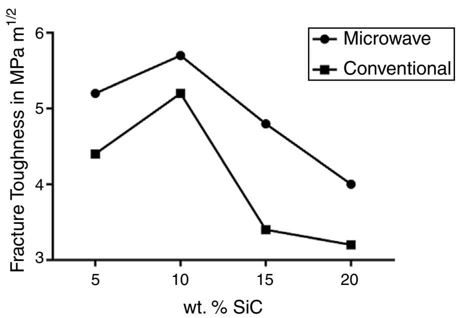

Figure 5 – Fracture toughness for the Al_2O_3/x wt.% SiC samples by both sintering methods.

increases the porosity and declines the density of the composite [28].

Fig. 5 indicates the fracture toughness versus SiC content. It shows that fracture toughness increases as the SiC content increases till 10 wt.% and then decreases with the SiC content up to 20 wt.%. When SiC content is less (5 and 10 wt.%), fracture toughness increases due to the toughening mechanism such as crack pinning. While the SiC content is more, the grain size becomes finer which reduces the fracture toughness. In general, grain size influences the toughening effect by grain-bridging, and if grain size is more, the fracture toughness will also be more [20]. In both the sintering methods, the fracture toughness shows the same phenomenon, however in microwave shows increased toughness because of uniform grain growth due to volumetric heating and ionic diffusion of the Al_2O_3 particle [25,26]. The average thermal expansion coefficient of Al_2O_3 ($8.1 \times 10^{-6}/^{\circ}C$) is about two times of SiC ($4.0 \times 10^{-6}/^{\circ}C$) and as a result residual internal stress is created in conventional method resulting in decline in the fracture toughness [29,30].

Phase variation and microstructural evolution

Fig. 6(a) and (b) illustrates the pattern of X-ray diffraction of the conventional and microwave-sintered samples. As it is seen, there is a SiO_2 phase formed in all four compositions produced by conventional sintering and very negligible amount of SiO_2 in 15 and 20 wt.% SiC composites produced by microwave sintering due to low sintering time than conventional sintering [31]. In both the methods, the intensity of peak increases with increase in the SiC phase. By comparing X-ray diffraction patterns, the only considerable point is increasing of peak's

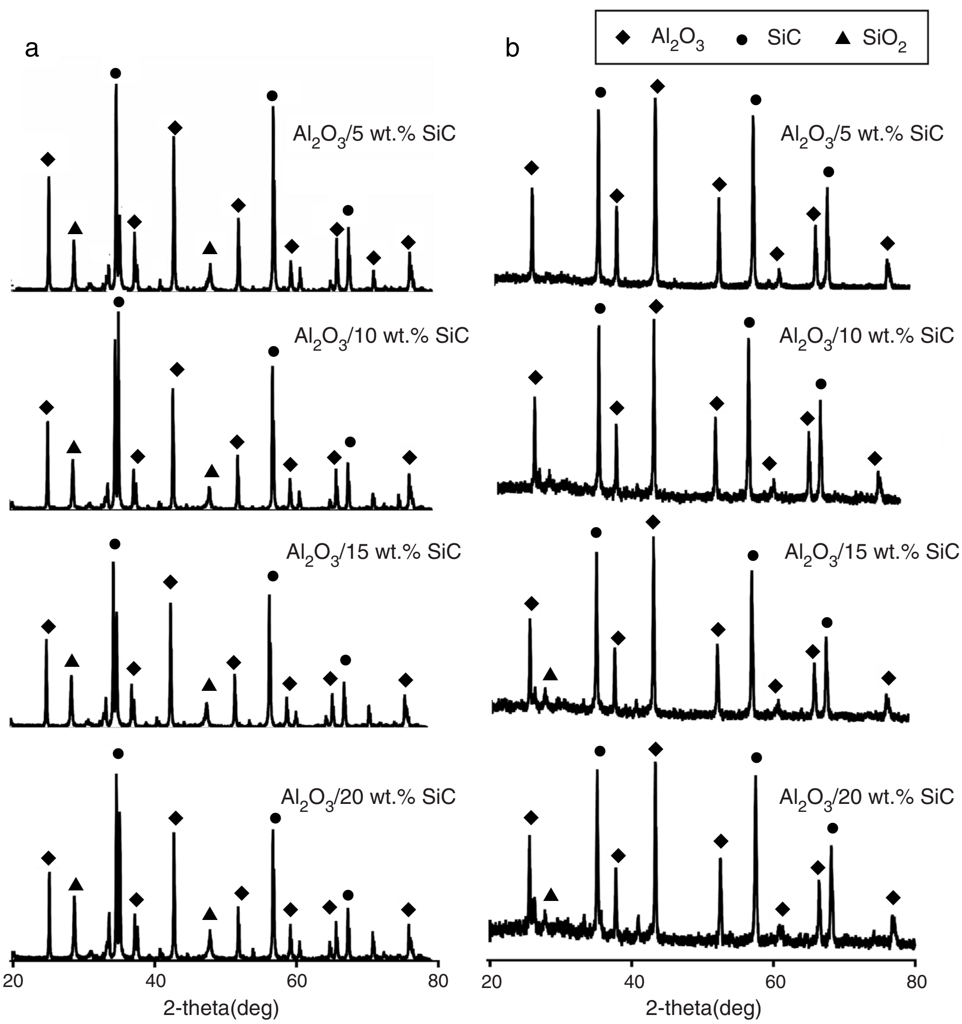

Figure 6 – The XRD pattern for the (a) conventional sintered samples and (b) microwave-sintered samples.

Table 2 – Crystalline size by Scherrer's method.

Samples	Crystalline size (nm)			
	Conventional sintering		Microwave sintering	
	Al_2O_3	SiC	Al_2O_3	SiC
Al_2O_3/5 wt.% SiC	84.08	71.10	70.23	67.32
Al_2O_3/10 wt.% SiC	78.16	67.90	49.72	45.39
Al_2O_3/15 wt.% SiC	87.77	83.04	58.52	49.61
Al_2O_3/20 wt.% SiC	89.71	72.57	42.15	38.96

intensity and decreasing of peak's width in the microwave-sintered sample than in the conventional sintered sample. Table 2 shows the crystalline size of both Al_2O_3 and SiC phases in nanometers calculated by the Scherrer method. The crystalline sizes are within the range of 78–89 and 68–83 nm for Al_2O_3 and SiC phases, respectively, in the conventional sintered samples, whereas in the microwave-sintered samples the crystalline size range was 42–70 and 38–67 nm for Al_2O_3 and SiC phases, respectively. To conclude, the crystalline size of the microwave-sintered samples is finer than the conventionally sintered sample due to less sintering time.

Fig. 7 shows the microstructure evolution and morphological comparison with same magnification for all four compositions of the Al_2O_3/SiC ceramic composites prepared by conventional sintering. The microstructure indicates that the voids increases with increase in the SiC content, and a large number of voids are observed in Fig. 7(c) and (d) than in Fig. 7(a) and (b) which concedes the result discussed in density parameter. Fig. 7(a) and (b) shows the agglomeration of Al_2O_3 because of the less number of intergranular SiC particles. A glassy prismatic pattern was seen in the 10 wt.% SiC sample and increased in the 15 wt.% SiC sample, but prismatic bright formations are reduced in the 20 wt.% SiC sample. This indicates that glassy Al_2O_3 formation increases with SiC content up to 15 wt. % of SiC and then it decreases for 20 wt.% SiC [32].

Fig. 8 shows the microstructures of the microwave-sintered samples. It was evident that the voids are increased with SiC content but lesser than conventional sintering due to

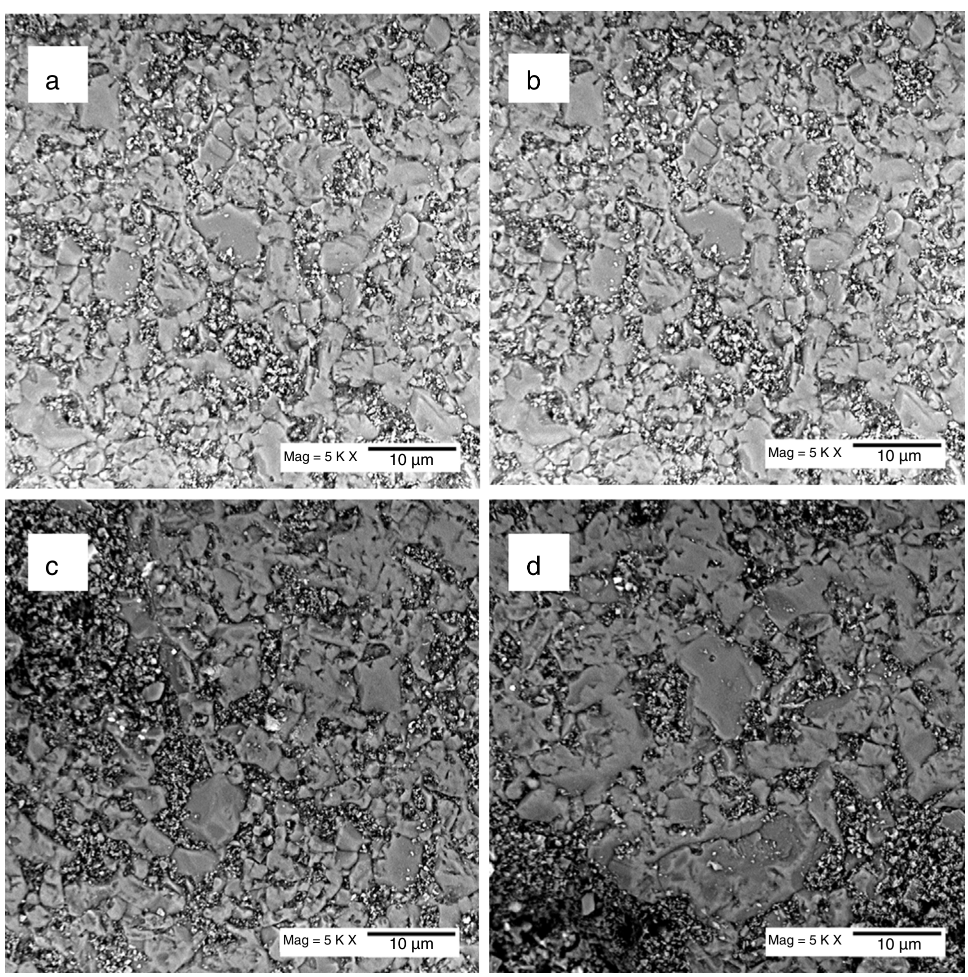

Figure 7 – The SEM figures of the conventional sintered Al_2O_3/x wt.%SiC samples: (a) 5 wt.% SiC; (b) 10 wt.% SiC; (c) 15 wt.% SiC and (d) 20 wt.% SiC.

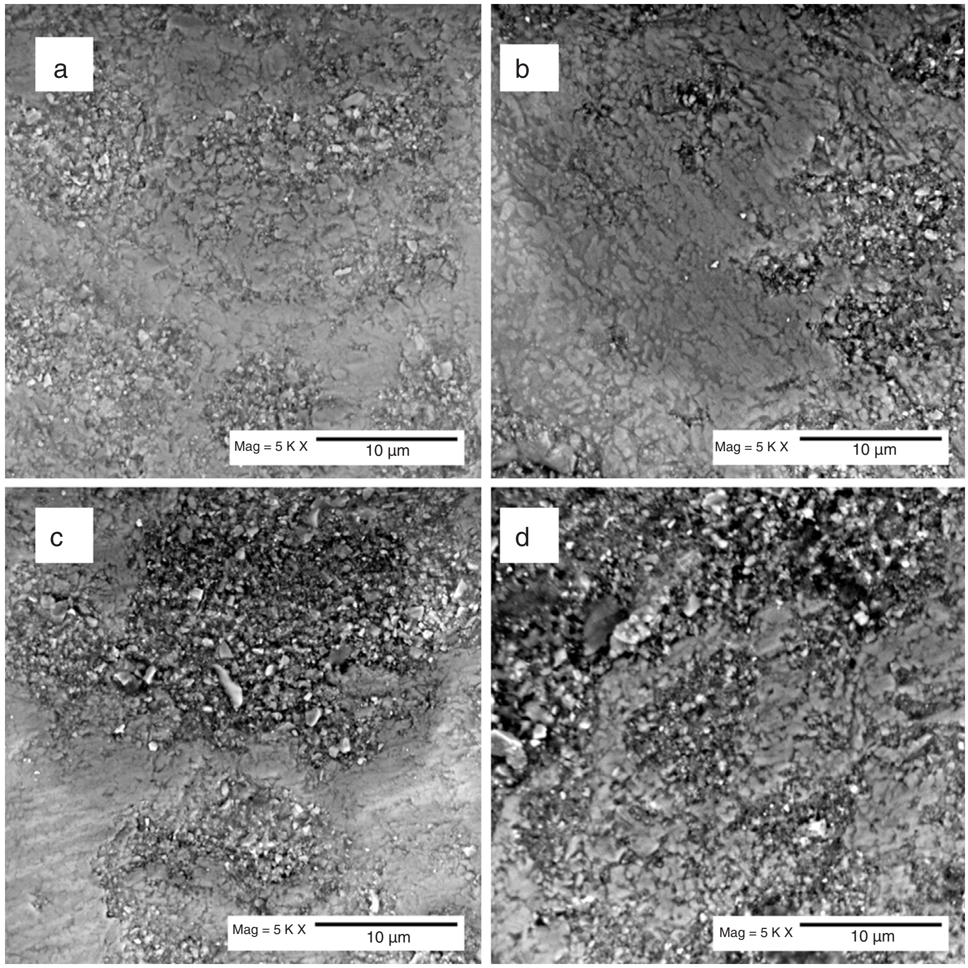

Figure 8 – The SEM figures of the microwave-sintered Al_2O_3/*x* wt.% SiC samples: (a) 5 wt.% SiC; (b) 10 wt.% SiC; (c) 15 wt.% SiC and (d) 20 wt.% SiC.

microwave effect. Fig. 8(a) and (b) shows the cluster compact formation of Al_2O_3 and glassy prismatic structure is shown in Fig. 8(c) only. From microstructural analysis, it was evident that density of the Al_2O_3/SiC ceramic composite increases when prepared by using microwave sintering than conventional sintering. It is interesting to note that, while comparing microstructures of both the conventional and microwave sintering methods, uniform agglomeration is formed more in microwave sintered than in the conventional sintered samples [25,26,30].

Conclusion

- Al_2O_3–SiC ceramic composites containing different weight fraction (5, 10, 15 and 20 wt.%) of SiC were prepared by both the conventional and microwave sintering methods. Comparative studies on mechanical properties and microstructures between microwave sintering and conventional sintering were done.
- The density of the Al_2O_3–SiC composite increases for 5 wt.% SiC in both the methods, and then decreases with further increase in SiC content because the presence of SiC particles blocks the grain boundary movement. In all the cases, microwave sintering shows higher density than conventional sintering due to volumetric heating and shorter sintering time.
- Matrix grain size decreases with an increase in SiC content for both the methods, because of the presence of intergranular SiC particles which produce less grain boundary pinning. Uniform grain formations formed in microwave sintering than in conventional sintering.
- Higher hardness of 24.6 and 22.6 GPa were obtained for the 10 wt.% SiC samples by microwave and conventional sintering, respectively, because of the presence of hard secondary SiC phase. In this case, SiC content increases above 10 wt.% hardness decreases due to decrease in densification. In all compositions, higher hardness was obtained in microwave sintering than conventional sintering, because the internal residual stress caused due to thermal expansion declines the hardness in conventional sintering method.
- Fracture toughness also increases up to 10 wt.% SiC and decreases with further addition of SiC content. Higher

fracture toughness of 5.7 and 5.2 MPa m$^{1/2}$ obtained for Al_2O_3/10 wt.% SiC by microwave and conventional sintering method, respectively. Finer grain size reduces the fracture toughness due to grain-bridging. Microwave shows enhanced fracture toughness due to uniform grain formation.

- The X-ray diffraction pattern shows the formation of SiO_2 phase in conventional sintering and only negligible SiO_2 phase in microwave sintering due to less sintering time. The crystalline sizes of the microwave-sintered samples are finer than the conventional sintered samples, due to less sintering time in microwave sintering.
- Microstructures reveal the increase in voids with increase in SiC content in both the methods. But, uniform agglomeration was present in the microwave-sintered samples than the conventional sintered samples, because of microwave effect and less sintering time.
- The result shows that the presence of secondary-phase SiC particles with microwave sintering method enhances the properties of Al_2O_3–SiC ceramic composites than conventional sintering method.

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