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Research Article

A Comparative Study on Crack-Healing Ability of Al_2O_3/SiC Structural Ceramic Composites Synthesized by Microwave Sintering and Conventional Electrical Sintering

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This study was conducted to assess and compare the crack-healing ability of conventional electrical sintered and microwave sintered Al_2O_3/x wt. % SiC ($x = 5$, 10, 15, and 20) structural ceramic composites. The crack-healing ability of both conventional electrical sintered and microwave sintered specimens was studied by introducing a crack of ~100 μm length by Vickers's indentation and conducting a heat treatment at 1200°C for dwell time of 1 h in air. The flexural or bending strength of sintered, cracked, and crack-healed specimens was determined by three-point bending test, and the phase variations by X-ray diffraction and SEM micrographs before and after crack-healing of both the sintering methods were studied and compared. The results show that almost all the specimens recovered their strength after crack-healing, but the strength of microwave sintered Al_2O_3/SiC structural ceramic composites has been shown to be better than that of conventional electrical sintered Al_2O_3/SiC structural ceramic composites. The microwave sintered crack-healed Al_2O_3/10 wt. % SiC specimen shows higher flexural strength of 794 MPa, which was 105% when compared with conventional electrical sintered Al_2O_3/10 wt. % SiC and crack-healed Al_2O_3/10 wt. % SiC specimen. It was found by X-ray diffractogram that before crack-healing, all the conventional electrical sintered samples have SiO_2 phase which reduce the crack-healing ability and microwave sintered samples with 15 and 20 wt. % SiC show lesser SiO_2 phase and 5 and 10 wt. % SiC samples have no SiO_2 phase before crack-healing. However, after crack-healing treatment, all the samples have distinct SiO_2 phase along with Al_2O_3 and SiC phases. Microwave sintered Al_2O_3/10 wt. % SiC specimen cracks were fully healed which was evident in SEM micrographs.

1. Introduction

In recent years, more concentration has been given to the structural ceramics with exemplary mechanical, chemical, and thermal properties [1]. However, structural ceramics are responsive to the existence of surface cracks by reason of their brittle nature. Moreover, steadfastness of structural ceramics considerably declined because of crack growth during machining or in service [2]. Crack-healing might be an effective way to overcome the effects of cracks and to recuperate the strength of structural ceramics. During the past decades, crack-healing nature of various ceramic composites by heat treatment was researched by the material researchers. Gupta [3] synthesized thermally shocked MgO and observed the crack-healing ability from 1400°C to 1650°C. Nakao et al. [4] found excellent crack-healing ability in Al_2O_3/30 vol% SiC composite, mullite/15 vol% SiC composite, and Si_3N_4/20 vol% SiC composite when heat treated at 1200°C for 1 hour dwell in air. The cracks created by machining in Al_2O_3, Si_3N_4, and mullite ceramics reinforced with SiC could be healed at 1200°C, 1300°C, and 1300°C, respectively, with the dwell time of 1 hour in air [5]. Lee et al. [6] showed that the indentation crack of ~100 μm was healed by conducting a healing treatment at 1300°C for 1 hour in air including a second phase SiC particles into Sc_2O_3 and AlN. Nam and Hwang [7] found that the optimal crack-healing condition for ZrO_2/SiC was 800°C for 5 hours in air.

Ando et al. [8] and Takahashi Koji et al. [9] found that the SiC present in Si_3N_4/SiC ceramic composite has the ability to heal the surface crack of length ~100°μm at 1200°C or 1300°C temperature with a dwell time of 1 h in air. Nakao et al. [10] observed the crack-healing in Al_2O_3/SiC whiskers/SiC particles ceramic composite and Al_2O_3/SiC whiskers ceramic composite at 1300°C with 1 hour soaking time in air due to the formation of SiO_2 by the oxidation of SiC phase and healing of the surface crack length below 250 μm and below 200 μm, respectively. The SiO_2 formed by oxidation of SiC was bonded with the crack surface, and crack-healing was achieved with a soaking time of 1 hour in air.

Later, material researchers investigated the crack-healing ability of high dense Al_2O_3/SiC ceramic composite synthesized by hot pressing sintering. However, formation of SiO_2 phase during sintering of ceramics depreciates the healing ability, as well as it affects the quality of the material. Mandal et al. [11] found that ~32% of SiC gets oxidized and converted into SiO_2 and depreciates the product quality significantly during the conventional sintering process. To overcome this limitation, structural ceramics are synthesized by using microwave heating because it elicits very low SiO_2 formation than other conventional sintering process. Microwave sintering is viewed as a prevailing method than conventional sintering processes because it can provide enhanced mechanical properties and better finer microstructure with minimum energy consumption [12, 13]. Microwave sintering has been successfully used to synthesize Al_2O_3-based structural ceramics with constructive mechanical properties [14–22]. In order to produce crack-healing in Al_2O_3/SiC structural ceramic composites, the presence of SiC phase after sintering process was very much needed. As per above investigation, crack-healing was mostly in need of the formation of SiO_2 during healing process. In this study, crack-healing ability of conventional electrical sintered and microwave sintered Al_2O_3/x wt.% SiC (x = 5, 10, 15, 20) ceramic composite materials was compared.

2. Experimental Procedure

α-Al_2O_3 particles (average grain size = 3 μm, 99.5% purity, sigma Aldrich) and β-SiC particles (average grain size = 1 μm, 99% purity sigma Aldrich) were used as the starting materials. To advance the homogeneity of the raw materials, the mixtures of Al_2O_3 with different wt.% of SiC (5, 10, 15, and 20) were ball milled along with isopropyl alcohol medium using WC ball at 300 rpm for 6 hours. After milling, the homogeneous mixture was desiccated at 80°C and sieved through 200 mesh. All the four homogeneous powders were compacted into square plate (45 mm × 45 mm × 3 mm) in uniaxial cold press at 60 MPa for 30 seconds discretely. One set of plates was sintered in an electric resistance heating furnace with $MoSi_2$ as heating elements under the following condition: temperature = 1500°C; heating rate = 10°C per minute; and dwell time = 300 minutes. One more set of plates were sintered in a microwave furnace with 2.45 GHz, and it consists of magnetron as the microwave source element with SiC-based susceptor materials as supplementary heating elements under the following condition: temperature = 1500°C, holding time = 15 minutes, and input power ranging from 0.9 to 2.4 kW. Both sintering processes use an air atmosphere. A noncontact type infrared sensor was used to measure the temperature in microwave furnace and Eurotherm (Model 2416) microprocessor-based PID controller with digital indicator was used to control the temperature. In both the sintering methods, conventional electrical furnace sintering and microwave sintering method, specimens were cooled in the furnace itself. The sintered plates were cut into rectangular bar (45 mm × 4 mm × 3 mm) specimens. One face of the specimen was well polished using diamond paste in lapping machine. A semielliptical crack of ~100 μm was made on two set of conventional electrical sintered and microwave sintered specimens at the centre by Vickers indenter with a load of 29.4 N. From the review article [23], the optimum condition for crack-healing an Al_2O_3/SiC ceramic composite to recover its full strength is annealing at 1200°C for 60 minutes in air. Therefore, one set of cracked specimens was subjected to crack-healing treatment in an electric resistance heating furnace with $MoSi_2$ as heating elements and another set was kept as cracked for investigation. The flexural strength of smooth, cracked, and crack-healed specimens was determined by the three-point bending test based on the ASTM C1161 standard with a support span (L) of 40 mm length with a speed of 0.5 mm/min. Three specimens were used in each condition to calculate the flexural strength. Figure 1 shows the schematic diagram of three-point bending test, and the flexural strength was calculated using the following equation:.

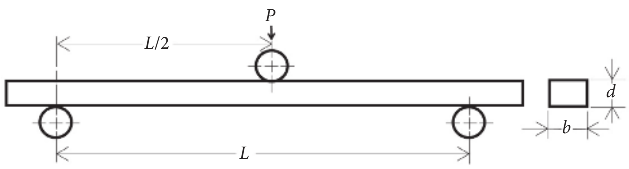

FIGURE 1: Schematic diagram of the three-point bending test.

$$S = \frac{3PL}{2bd^2}, \quad (1)$$

where P is the break force, L is the outer (support) span, b is thespecimen width, and d is the specimen thickness.

X-ray diffraction (XRD-SmartLab, Japan) was used to study the various phase composition of smoothed and crack-healed specimen synthesized by both the conventional electrical sintering and microwave sintering method. The X-ray diffraction was done using Cu–K beta radiation with 30 mA electric current and 45 kV accelerated voltage. The micrographs of cracks before and after healing were scanned by scanning electron microscopy (FESEM, Supra-55, Carl Zeiss, Germany).

3. Result and Discussion

3.1. Microstructure. Figure 2 shows the microstructure and morphological of Al_2O_3 with four different wt.% of SiC composites synthesized by conventional electrical sintering. As a result of the presence of more intergranular SiC particles in samples with 15 and 20 wt. % SiC (Figures 2(c) and 2(d)), the voids are more than 5 and 10 wt. % SiC (Figures 2(a) and 2(b)) samples. Figures 2(a) and 2(b) confirm that cluster of Al_2O_3 is more than that in Figures 2(c) and 2(d) because of lesser amount of SiC particles.

Microstructures of microwave sintered Al_2O_3/SiC ceramic composite samples are shown in Figure 3. It was noticeable that with increase in SiC content, the voids are increased but microwave effect reduces the voids by increasing the density. Figures 3(a) and 3(b) show clump of dense Al_2O_3 phase, and Figure 3(c) shows glassy prismatic structure. It is fascinating to notice that uniform agglomeration is noticed more in the microstructures of microwave sintered Al_2O_3/SiC ceramic samples than microstructures of conventional sintered Al_2O_3/SiC ceramic samples.

3.2. Flexural Strength. The relationship between the wt. % of SiC in Al_2O_3/SiC ceramic composite and the flexural strength of smoothed, precracked, and crack-healed specimen synthesized by conventional electrical sintering and microwave sintering is given in Table 1. In all the cases, the flexural strength of microwave sintered specimen was more than that of the conventional electrical sintered specimen, indicating that microwave heating enhances the flexural strength by grain refinement which was evident in the microstructure. The flexural strength of crack-healed specimens of both the conventional electrical and microwave sintering is higher than that of the smooth specimens of both the methods. Likewise, when comparing the flexural strength of cracked specimens of both the methods with that of the crack-healed specimens of both conventional electrical and microwave sintered methods, crack-healed samples show higher flexural strength than cracked specimens. This indicates that all specimens recovered flexural strength and showed that the cracks were healed partially or completely. The higher flexural strength of 794 MPa was obtained for microwave sintered crack-healed Al_2O_3/10 wt.% SiC structural ceramic composite. The flexural strength of crack-healed Al_2O_3/10wt. % SiC sample synthesized by the microwave sintered method was improved up to 105%, when correlated with flexural strength crack-healed Al_2O_3/10 wt. % SiC specimen synthesized by conventional electrical sintering. Restoring full strength through oxidation has also been suggested by Koji Takahashiet al. [6] and Nakao et al. [10]. They synthesized ceramic composites based on Al_2O_3 and found that the inclusion of secondary-phase SiC particles improves crack-healing through oxidation and restores flexural strength.

3.3. Phase Variation after Crack-Healing. XRD pattern of conventional electrical sintered and microwave sintered Al_2O_3/SiC ceramic materials before and after crack-healing heat treatment at 1200°C with a dwell time of 1 h in air is shown in Figure 4. XRD patterns of all the samples before and after the crack-healing heat treatment show Al_2O_3 and SiC as two main phases. After crack-healing heat treatment process, the clear sharp peaks of SiO_2 were detected in the diffraction pattern of all the samples, indicating that Al_2O_3/SiC ceramic material gets suffered by oxidation. The reaction due to oxidation of SiC by crack-healing heat treatment for the formation of new phases could be described in the following equation:

$$SiC + \frac{3}{2}O_2 \longrightarrow SiO_2 + CO. \quad (2)$$

However, XRD pattern of conventional electrical sintered samples shows clear sharp peaks of SiO_2 phase before the crack-healing heat treatment and some smaller peaks in microwave sintered Al_2O_3/20 wt. % SiC sample. XRD pattern before and after crack-healing reveals that amount of SiC particles available for crack-healing reaction was more in samples synthesized by microwave sintering than samples synthesized by conventional electrical sintering. SiC phase was still detectable in the samples after crack-healing because SiC particles on the surface and cracks are only exposed to air and get oxidized into SiO_2 and the SiC particles within the surfaces are not getting oxidized. In previous studies, it was also observed that a SiO_2 phase forms in all four traditional sintering compositions, but only a small amount of SiO_2 forms in 15 and 20 wt.% of SiC composites synthesized by microwave sintering due to the shorter sintering time [19, 20].

3.4. SEM Micrographs. SEM micrographs of radial cracks produced by Vickers indentation in conventional electrical sintered Al_2O_3/SiC structural ceramics before and after

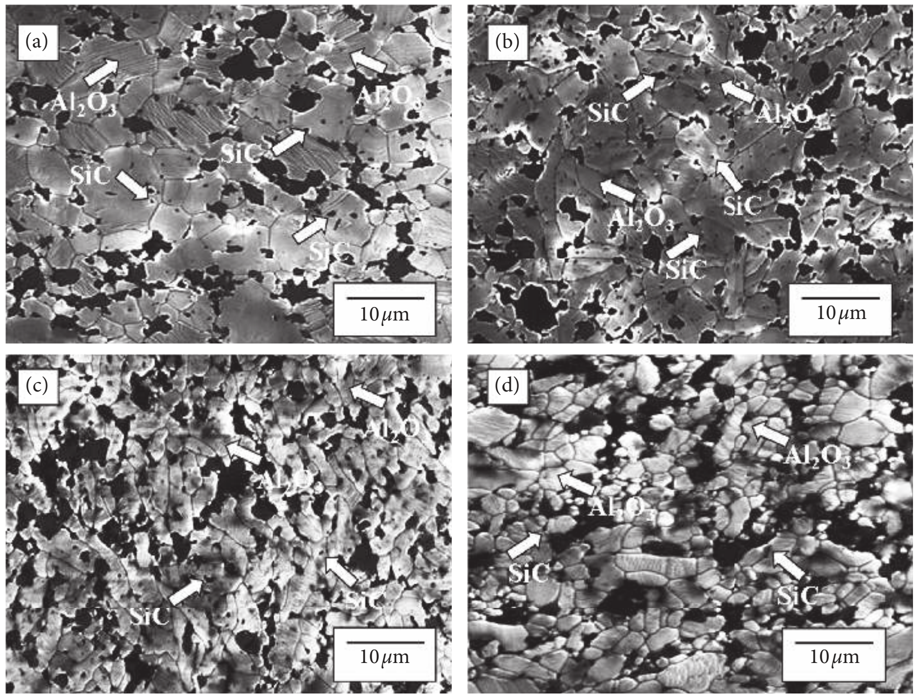

FIGURE 2: SEM figures of conventional electrical sintered Al_2O_3/xwt.% SiC samples: (a) 5 wt. %SiC; (b) 10 wt.%SiC; (c) 15 wt.%SiC; (d) 20 wt.%SiC.

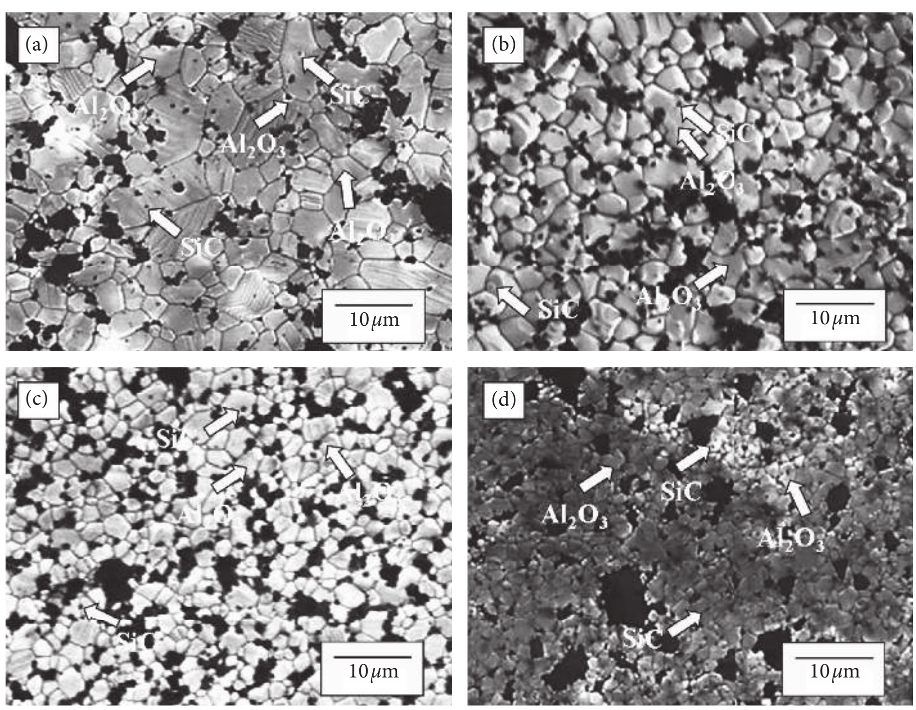

FIGURE 3: SEM figures of microwave sintered Al_2O_3/x wt.%SiC samples: (a) 5 wt. % SiC; (b) 10 wt.% SiC; (c) 15 wt.% SiC; (d) 20 wt.% SiC.

crack-healing are shown in Figure 5. After crack-healing heat treatment at 1200°C for 1h in air, the surface of specimen became rough due to the oxidization of the particles. Healing occurs in the radial cracks produced by Vickers indentation which is shown in Figure 5. However, the radial cracks are not healed for the entire length, and the cracks with minimum width are healed completely in all the conventional electrical sintered samples. This incomplete healing in conventional electrical sintered sample is primarily because of the nonavailability of more SiC secondary-phase particles in the cracked region. The reason for this was oxidation of SiC particles into SiO_2 during the sintering of

Table 1: Flexural strength of smooth, cracked, and crack-healed conventional electrical sintered and microwave sintered Al_2O_3/SiC ceramic composites.

Condition	Wt. % of		Flexural strength σ in MPa	
	Al_2O_3	SiC	Conventional electrical sintering	Microwave sintering
Smooth specimen	95	5	302 ± 23	318 ± 32
	90	10	592 ± 34	624 ± 38
	85	15	512 ± 18	543 ± 28
	80	20	467 ± 42	508 ± 18
Cracked specimen	95	5	212 ± 38	203 ± 33
	90	10	242 ± 26	257 ± 42
	85	15	287 ± 18	312 ± 18
	80	20	262 ± 28	315 ± 22
Cracked + healed	95	5	427 ± 32	488 ± 41
	90	10	758 ± 24	794 ± 32
	85	15	647 ± 14	658 ± 34
	80	20	596 ± 21	623 ± 26

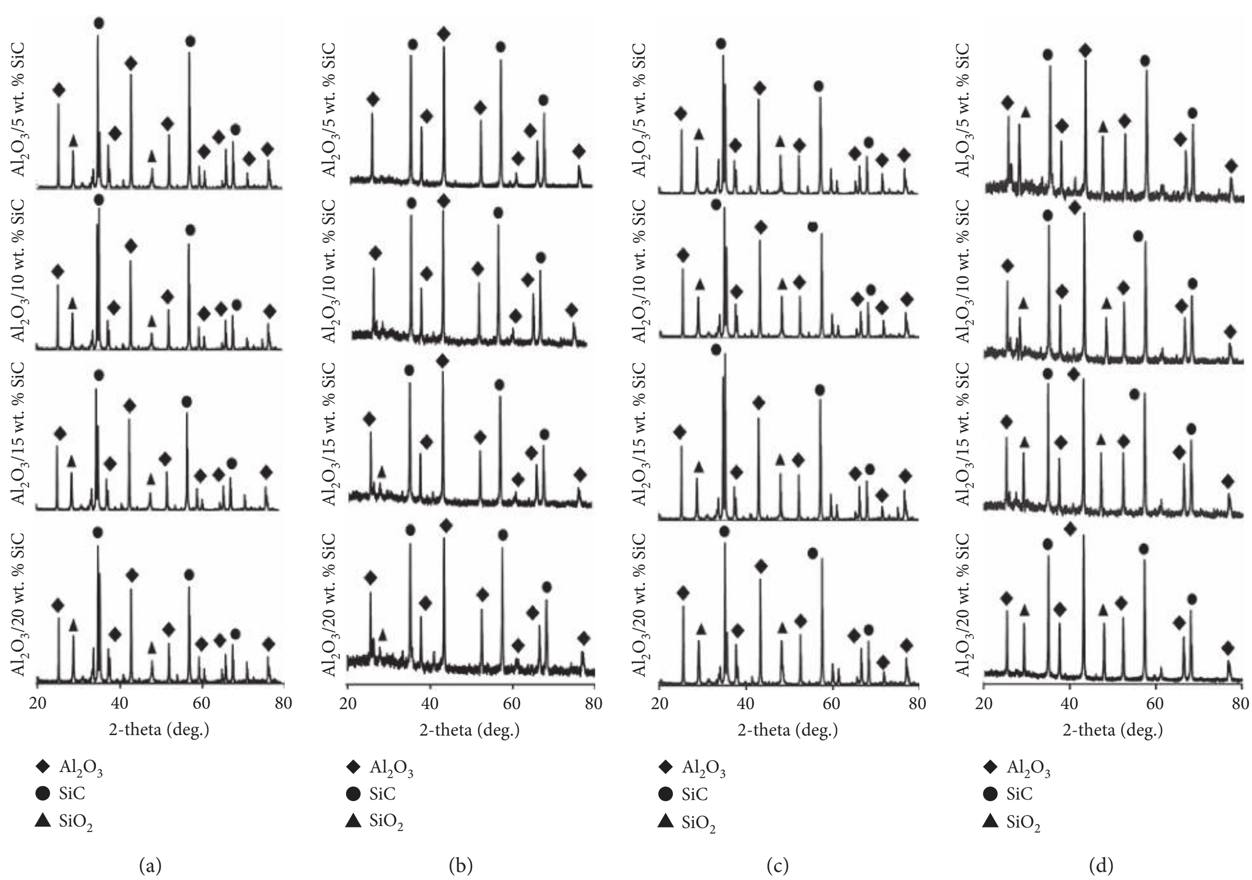

Figure 4: XRD pattern of Al_2O_3/x wt.% SiC: (a) conventional electrical sintered sample; (b) microwave sintered sample; (c) crack-healed conventional electrical sintered sample; (d) crack-healed microwave sintered sample.

green consolidated sample in conventional electrical sintering, which was evident by the detection of clear SiO_2 peaks in the XRD patterns of all four composition before the crack-healing process. The oxidation of SiC particles can be reduced by minimizing the holding time during the sintering process, but this will cause the reduction in the density of Al_2O_3/SiC structural ceramics synthesized by conventional electrical sintering.

Figure 6 shows the SEM micrographs of radial cracks produced by Vickers indentation in microwave sintered Al_2O_3/SiC structural ceramics before and after crack-healing. Similar to conventional electrical sintered samples, after crack-healing heat treatment at 1200°C for 1h dwell time in air, the surface of specimen became rough due to oxidation of the particles. Because of the less SiC content in the ceramic structure, the radial cracks are not healed for the entire

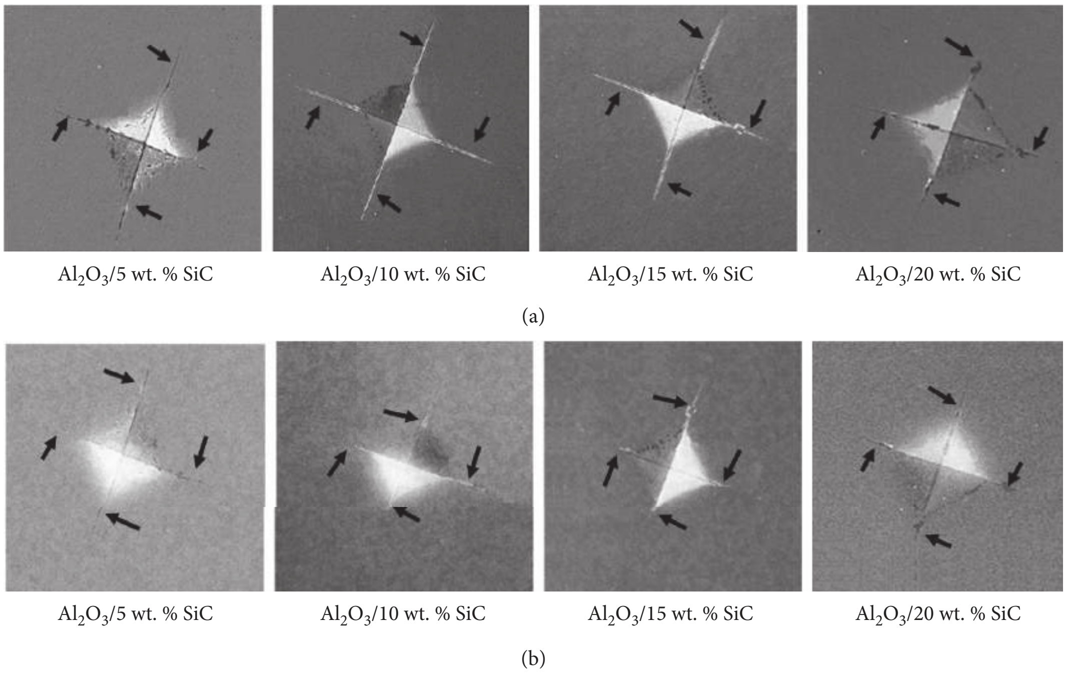

Figure 5: SEM micrographs of radial cracks by Vickers indentation in conventional electrical sintered Al_2O_3/SiC structural ceramics (a) before crack-healing and (b) after crack-healing.

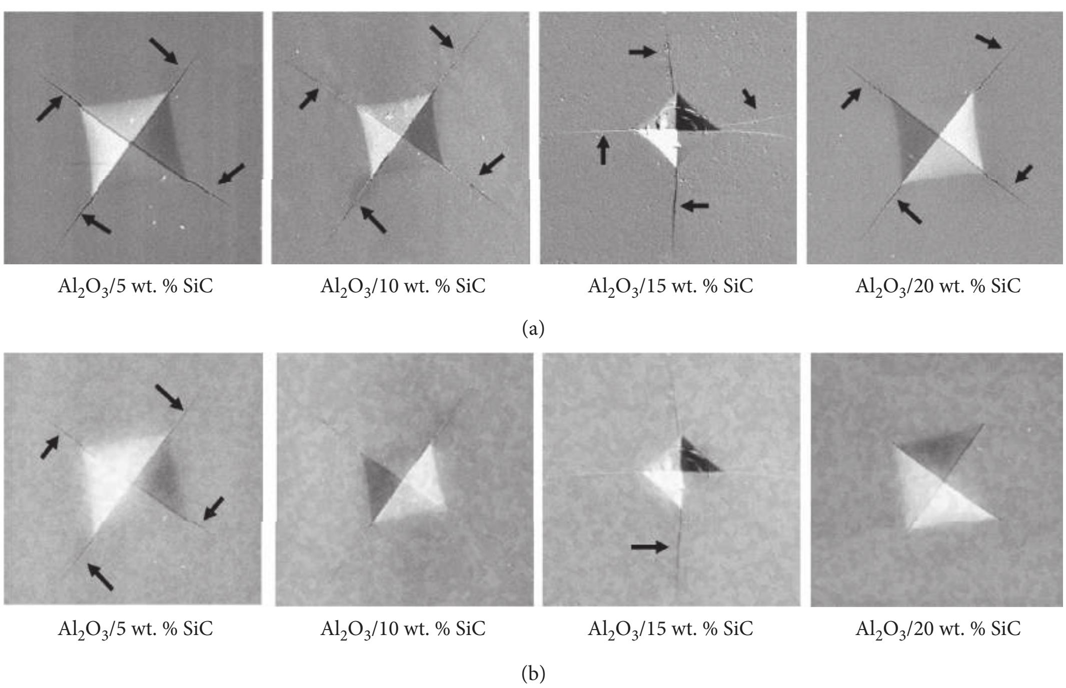

Figure 6: SEM micrographs of radial cracks produced by Vickers indentation in microwave sintered Al_2O_3/SiC structural ceramics (a) before crack-healing and (b) after crack-healing.

length in Al_2O_3/5 wt.% SiC structural ceramics which is indicated in Figure 6 by arrow marks in after crack-healing. Mostly, all the radial cracks are healed in others Al_2O_3/SiC ceramic composites with 10, 15, and 20 wt.% SiC. This complete healing was due to the availability of more SiC particles after the microwave sintering. The oxidation of SiC particles was very negligible in microwave sintering because of its volumetric heating and lesser holding time. Due to this, crack-healing ability of microwave sintered samples increases than conventional electrical sintered sample. In the

SEM micrograms of both conventional electrical sintered and microwave sintered samples after crack-healing, spongy appearance is present all over the surface due to the oxidation of SiC particles, and the surface after crack-healing heat treatment become rough when compared with sample surface without crack-healing heat treatment.

4. Conclusion

In this research, crack-healing ability of conventional electrical sintered and microwave sintered Al_2O_3/x wt.% SiC ($x = 5$, 10, 15, and 20) ceramic composites was discussed and compared. For this, the crack length of approximately 100 μm was produced by the Vickers indentation method. The crack-healing condition of 1200°C for 1h in air was used, which was identified from the review.

(i) SEM microstructures reveal that with increase in SiC wt.%, the voids also increase in the samples synthesized by both the conventional electrical sintering and microwave sintering methods. Due to volumetric heating and less sintering time in microwave sintering process, the samples synthesized by this method show uniform agglomeration than samples synthesized by the conventional electrical sintering method.

(ii) The higher flexural strength of 794 MPa was obtained for microwave sintered crack-healed Al_2O_3/10 wt. % SiC structural ceramic composite, which was 105% when compared with conventional electrical sintered crack-healed sample. In all the cases, microwave sintered sample is superior than conventional electrical sintered sample. The improvement in flexural strength after healing in microwave sintered Al_2O_3/SiC sample might have resulted from the availability of more SiC particles than conventional electrical sintered Al_2O_3/SiC sample because, during the synthesize of ceramic material by conventional electrical sintering, SiC particles get oxidized into SiO_2. This will reduce the crack-healing ability and amount of strength recovery when compared with microwave sintered Al_2O_3/SiC ceramics.

(iii) The two main phases detected before and after the crack-healing in XRD pattern are Al_2O_3 and SiC phases. After crack-healing heat treatment process at 1200°C for 1°h dwell in air, the SiC particles in the samples get oxidized and form SiO_2 phase and it was clearly visible by the sharp peaks in the diffractogram.

(iv) At 1200°C for 1 hour dwell in air, the crack-healing mechanism was attributed by the formation of SiO_2 phase in Al_2O_3/SiC structural ceramic materials. Results of microstructure analysis indicated that the crack was healed completely in microwave sintered Al_2O_3 with 10, 15, and 20 wt. % SiC by the oxidized product. In all other cases, the radial cracks are partially healed, but strength was recovered when compared with smooth specimen.

Data Availability

The data used to support the findings of this study are included within the article.

Disclosure

It was performed as a part of the Employment of Addis Ababa Science and Technology University, Addis Ababa, Ethiopia.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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