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On the Role of Sintering on Microstructural Development of Alumina Toughened Nanocomposites

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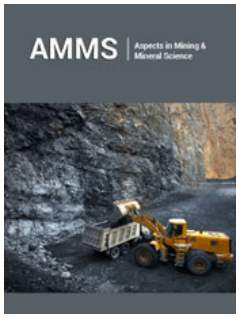
ISSN: 2578-0255



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Submission: October 11, 2022
Published: November 11, 2022

Volume 10 - Issue 2

How to cite this article: Meena KL and Srivatsan TS*. On the Role of Sintering on Microstructural Development of Alumina Toughened Nanocomposites. Aspects Min Miner Sci. 10(2). AMMS. 000733. 2022.
DOI: 10.31031/AMMS.2022.10.000733

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Abstract

In this research paper, the physical properties, and microstructural characteristics of 3 mol % Yttria-Stabilized-Zirconia (3YSZ) matrix reinforced with 10 volume percent of alumina (Al_2O_3) is presented and briefly discussed. The composites were developed using the techniques of Conventional Sintering [1600 °C, 5 °C/minute and held for 6 hours] and Microwave Sintering [1600 °C, 25 °C/minute and held for 1 hour] under pressure-less condition. The sintered powders were subsequently compacted using a uniaxial cold isostatic press at a pressure of 200MPa. The relative density and average grain size of both the conventional sintered samples and microwave sintered samples were determined and found to be [density: 98.16±0.15, grain size: 600nm] and [density:99.29±0.10, grain size: 421nm], while the thermal conductivity was determined to be 2.3W/mK and 2.6W/mK, respectively. A near uniform microstructure was observed for both the samples. The microwave sintered samples were observed to have superior physical properties when compared one-on-one with the conventional sintered counterpart. This study provides useful information specific to the development of thermal barrier coatings and for use in dental applications.

Keywords: Microwave sintering (MW); Conventional sintering (CS); Alumina-toughened-zirconia (ATZ); Physical properties; Microstructure analysis

Introduction

The composite based on alumina-based ceramics offer an excellent combination of properties to include the following: (i) overall chemical inertness, (ii) high abrasion resistance, and (iii) high-hot-hardness against extreme environments [1]. Alumina (Al_2O_3) is often chosen for making cutting tool inserts in comparison to carbides and even the family of high-speed steel. This is essentially because of the overall inert behavior or response characteristics of alumina (Al_2O_3) upon exposure to an elevated temperature [2]. Alumina-based ceramic cutting inserts are currently being chosen for use in wear-critical applications to include the following: (i) mixers, (ii) grinders, (iii) coal chutes, and (iv) ball mills [3]. Zirconia Toughened Alumina (ZTA) ceramics, classified to be a new and emerging generation of ceramic, is noticeably popular since they can exhibit remarkably higher [4 times] and fracture toughness when compared one-on-one with alumina [4]. Initially, the primary objective for the addition of Zirconia to alumina (Al_2O_3) was to improve its density. However, subsequent research studies have found and recorded Zirconia to be a potential reinforcing material capable of improving the fracture toughness of alumina (Al_2O_3) [5,6]. Overall, through the years several experiments have been conducted on the development of ZTA composites having an improved combination of properties. A few of these studies focused on Zirconia Toughened Alumina (ZTA) as the matrix material while few other studies were conducted with Zirconia Toughened Alumina (ZTA) as a viable reinforcement to other metallic matrices, such as the iron [7], aluminium [8] and even cast iron [9]. However, like most other materials, the ceramic composites tend to possess certain drawbacks and often suffer from issues specific to corrosion resistance and oxidation resistance both at room temperature and high temperatures. Besides, ceramics like zirconium oxide (ZrO_2) and aluminum oxide (Al_2O_3) do not suffer from these drawbacks. However, they

tend to become brittle when attempting to improve their strength thereby curtaining their selection and use for performance-critical applications. Further, the low cost of Zirconia and Alumina makes them attractive to both designers and manufacturers for selection and use in applications despite their inferior mechanical properties when compared one-on-one with other ceramics like the nitrides and carbides. Also, the important properties of alumina (Al_2O_3) like hardness, fracture toughness, flexural strength, and wear resistance can be improved by reinforcing it with zirconia [10-12].

Zirconia-based ceramics are a viable alternative to overcome the low fracture toughness of composites by a toughening mechanism [13]. To overcome the intrinsic brittleness of alumina (Al_2O_3), the yttria stabilized zirconia (YSZ) is added to an alumina matrix and the resultant Zirconia Toughened Alumina (ZTA) offers improved fracture toughness of the engineered composites by transforming the zirconia phase from tetragonal to monoclinic [14]. Pure zirconia favors the occurrence of catastrophic failure during cooling and while undergoing phase transformation from tetragonal to monoclinic [15]. The tetragonal phase of zirconia in the stable condition, without the use of reinforcements or dopants, can be obtained as fine particles that are smaller than a critical size [16]. The zirconia produces the monoclinic phase at room temperature, and a transformation phase from monoclinic to tetragonal when heated up to a temperature of 1700 °C, and with a structural change to the cubic phase when heated up to its melting point of 2716 °C [17]. After a critical review of the published literature, it was observed that several studies conducted on zirconia-based ceramic composites at a micro-scale on ceramic samples that were consolidated using the technique of conventional sintering, while only a few studies were recorded at the nanoscale. Recently, Oghbaei and co-workers [18] reported the use of Microwave Sintering (MW) process as a novel sintering technique when compared to conventional Sintering (CS), especially for materials that require sintering at high temperature. Further, microwave sintering (MW) also overcomes the drawbacks, such as: (i) poor and incomplete sintering [19], (ii) prolonged sintering times, and (iii) low heating rates [20], that are often associated with Conventional Sintering (CS).

The present study was focused on the development of alumina toughened zirconia (ATZ) composites by reinforcing alumina (Al_2O_3) to the Zirconia matrix and consolidating the composite powder using the techniques of Conventional Sintering (CS) and Microwave Sintering (MW). The consolidated samples were then systematically evaluated for their physical properties and microstructural characteristics. Also undertaken was a comparison of the phase analysis, microstructural characteristics, average grain size, relative density, microhardness, and fracture toughness of the developed composite samples.

Experimental Methods

Starting materials

Powders of high purity, i.e., 99% purity, 3 mol. pct. Yttria-Stabilized-Zirconia (3YSZ) with an average particle size of 40 nm and a monoclinic phase was used as the matrix material and procured from Zirox. Commercially available high purity (i.e., 99% purity] α-Al_2O_3 nanopowders, with an average particle size of 100nm, was used as the matrix material and procured from CUMI [Chennai, India]. The starting materials in the form of powder were used as high purity, thermally reactive type powders.

Development of the composites

The procedure and steps taken to produce nanocomposites used in this research study was to blend powders of alumina (Al_2O_3) with zirconia and consolidating the resultant mixture of powders using the techniques of Conventional Sintering (CS) and Microwave Sintering (MW) processes. Two separate ceramic composite samples were sintered using the approaches of Conventional Sintering [CS] and Microwave Sintering [MW] and essentially based on the elemental percentages provided in Table 1. For the purpose of clarity in the depiction of procedures for the development of desired composite material, a flow diagram showing all the steps and necessary details is shown in Figure 1. The reinforcement was 10 volume percent of Al_2O_3 added to the 3YSZ powders of the matrix powders and the resultant sample was sintered using the techniques of Conventional Sintering (CS) and microwave (MW) sintering. The starting powders were precision weighed and conformed to the amounts shown or provided in Table 1. The powders were then blended manually in an agate mortar using a pestle for full 40 minutes. Poly Vinyl Alcohol (PVA) was used to bind the powders particles together during blending. The binder helps in holding the powder particles together when they are pressed to get the required shape. This is essential since the green preforms are very delicate and consequently difficult to handle. The blended composite powder mixture was initially dried at 50 °C with the primary intent of removing any moisture present prior to sieving to remove all agglomerated lumps and impurities. A hydraulic machine was used to get a green compact cylindrical shape that measured 15mm in diameter and 3mm in height. This was made possible by using cold isostatic pressing (CIP: 200MPa) between a steel die and a punch. Zinc stearate was used as the solid lubricant to facilitate ease in ejection of the compacts, since clearance between the die and punch was only 10-20 microns. Also, the zinc stearate reduces the friction between the die and the punch. Polyvinyl alcohol (PVA) binder was used to avoid cracking that could occur in the sample during the sintering process. The "green" compact samples were subsequently sintered at a temperature of 1600 °C, using heating rates of (i) 5 °C/min, (ii) 25 °C/min and holding time of (a) 6 hours, and (b) 20 minutes, using the techniques of Conventional Sintering (CS) and Microwave (MW) sintering.

Table 1: Intended compositions for the Alumina Toughened Zirconia (ATZ) composite.

Developed ATZ Composite Through	Zirconia (vol. %)	Alumina (vol. %)	Sintering Temperature (°C)
CS	90	10	1600
MW	90	10	1600

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Figure 1: Flow diagram depicting the steps taken in engineering the composite material and the resultant study.

The conventional samples were sintered in a muffle furnace. For microwave (MW) sintering, a customized vacuum furnace having a 1.5kW power supply [Model: Omicron Scientific Equipment Company, Dwarka, New Delhi, India] was used to sinter the samples. The variations in temperature of the sample were measured using an infrared thermometer. The thermometer was focused on the surface through a small hole provided on the sintering chamber. In microwave (MW) sintering, the compacts were placed on a silicon carbide (SiC) susceptor, which acts as a hybrid heating source and then the susceptor along with the test sample was placed in the microwave furnace. Starting at room temperature, the temperature of the furnace gradually increased to the desire sintering temperature and held for some period prior to the initiation of cooling. The process parameters specific to Conventional Sintering (CS) and Microwave (MW) sintering are given in Table 2.

Table 2: Various process parameters and their ranges for both Conventional Sintering (CS) and microwave (MW) sintering.

Parameter	Conventional Sintering Process	Microwave Sintering Process
Pressure	200MPa (Hydraulic Pressure)	200MPa (Hydraulic Pressure)
Sintering Temperature	1600 °C	1600 °C
Heating rate	5 °C/min	25 °C/min
Holding time	6 hours	1 hour
Sintering Environment	Vacuum (10^{-6} torr)	Vacuum (10^{-6} torr)

Characterization methods

The samples were prepared for microstructure examination by initially grinding them on progressively fine grades of silicon carbide (SiC) impregnated emery paper followed by polishing on cloth until the samples achieved a near mirror-like surface finish. The polished samples were then chemically etched using hot phosphoric acid [H_3PO_4 at 250 °C] for 2 full minutes and gradually cooled to room temperature (25 °C) [21]. The as-polished and etched surfaces of the samples were observed in a field emission scanning electron microscope (FE-SEM). Selected samples were gold-coated prior to examination in the FE-SEM since they are non-conductive in nature. The 'Image J' software having a linear intercept approach program was used to calculate the average grain size of the engineered composite materials using the corresponding microstructure.

Results and Discussion

X-ray diffraction [XRD] analysis

An evaluation of the phases formed in the developed composites was done using X-Ray Diffraction (XRD) analysis and the corresponding XRD patterns are shown in Figure 2. From

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Figures 2a & 2b show peaks of the ATZ composites sintered using the techniques of Conventional Sintering (CS) and Microwave (MW) sintering. It is easily noticed that the major peaks indicate tetragonal zirconia (t-ZrO_2) [designated as t, ICDD files No. 072-7115] and alumina (α-Al_2O_3) [designated as α, ICDD files No. ICDD 089-7717], while the minor peaks of monoclinic zirconia (m-ZrO_2) [designated as m, ICDD files No. 037-1484] and cubic zirconia (c-ZrO_2) [designated as c, ICDD files No. 027-0997] were observed. There was no evidence of the presence of secondary phases. The highest peak intensity for the tetragonal t-ZrO_2 phase was detected at an angle of 30°. The formation and presence of secondary phases does affect both the microstructure and resultant properties of the developed composites. The metastable transformation of zirconia from t-ZrO_2 to m-ZrO_2 can be attributed to the 'local' stress that is induced in the field around a propagating crack that contributes to enhancing toughness of zirconia ceramics. The m-ZrO_2 phase in the Microwave (MW) sintered sample transformed to t-ZrO_2. Also, it was observed that the Microwave (MW) sintered sample did exhibit a lower amount of the monoclinic phase of ZrO_2. In the present study, despite selection of the same parameters, the intrinsic heating rate and cooling rate specific to both Conventional Sintering (CS) and Microwave (MW) sintering must be considered due essentially to salient differences in their operating mechanisms.

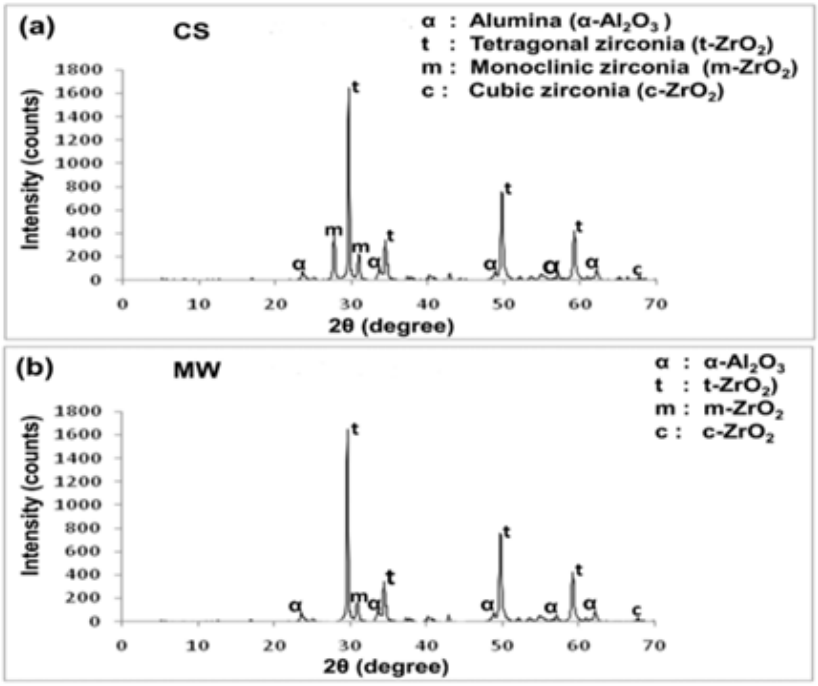

Figure 2: X-ray diffraction pattern showing peak intensities of Alumina Toughened Zirconia (ATZ) composite through Conventional Sintering (CS) and Microwave (MW) sintering.

Microstructural evaluation

An analysis of Scanning Electron Microscope (SEM) observation of the microstructures of composite materials developed by the addition of alumina (Al_2O_3) particulate reinforcements to the ZrO_2 matrix through both Conventional Sintering (CS) and Microwave (MW) sintering was carried out. The alumina grains (dark gray grains) were distributed homogeneously and well within the zirconia grains (small white grains) in the matrix as shown in Figure 3. Since the sintered samples were non-conductive in nature, they were coated with a thin gold layer prior to examination in a Scanning Electron Microscope (SEM). Figures 3a-3d shows different magnifications of the scanning electron micrographs for the Convention Sintered (CS) and Microwave (MW) sintered composites. From (Figure 3b), both white and dark grey phases of t-ZrO_2 and Al_2O_3, were easily observed as pointed by the arrows. The Microwave (MW) sintered sample was subject to rapid heating under pressure-less conditions. Both dispersion and densification of the powder particles occurred rapidly and did not get enough time for grain growth resulting in smaller grains when compared to the Conventional Sintered (CS) samples. The developed microwave (MW) sintered samples revealed a near uniform distribution in the matrix, and the grains were closely packed in the matrix with evidence of grain refinement and resultant dense composites. From (Figure 3a) it is observed that even though a near uniform distribution of the reinforced particles was achieved, the grain size was not as significant as that for the microwave (MW) sintered composite samples despite using the same sintering temperature. However, from (Figure 3a-3d), it is noticed that all the samples attained full density with homogeneously dispersed grains of Al_2O_3 and ZrO_2 in the alumina matrix. The rapid heating experienced by the microwave (MW) sintered samples does not give sufficient time for the occurrence of grain growth. The microwave (MW)

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sintered samples revealed a fully dense microstructure with fine grains of ZrO_2 uniformly dispersed through the matrix of the composite. The average grain size was measured by calculating the average value of both length and width of 10 random grains in the microstructure. The average grain size of the Conventional Sintered (CS) and microwave (MW) sintered samples was found to be 600±50nm and 421±30nm. The microwave (MW) sintered sample experienced rapid heating, suppression of grain growth with a resultant reduction in the average grain size, which led to an improvement in surface finish of the Alumina Toughened Zirconia (ATZ) sample. Also, no cracking was observed for the microwave (MW) sintered sample.

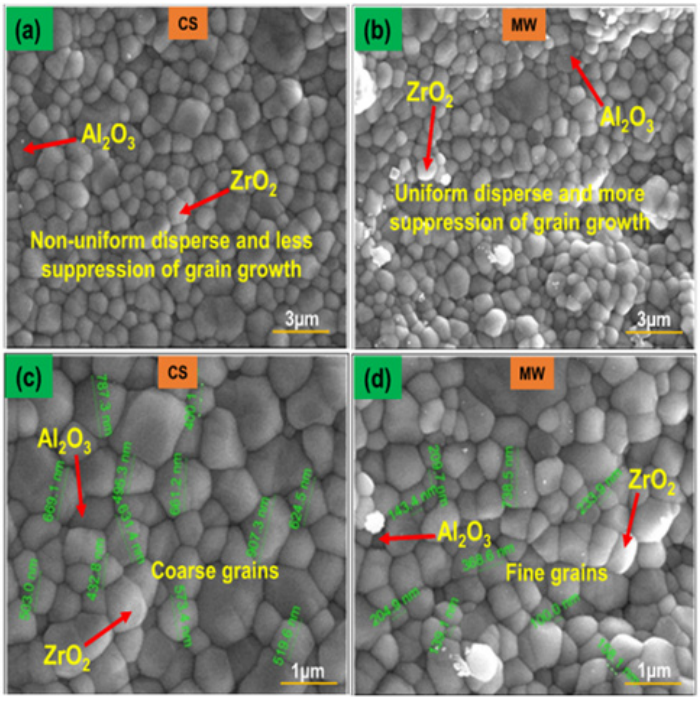

Figure 3: The thermally etched scanning electron micrographs showing microstructures of the developed Alumina Toughened Zirconia (ATZ) composites at 1600 °C, at
(i) A lower magnification (a) Conventional Sintering, and (b) Microwave sintering, and
(ii) A higher magnification (c) Conventional Sintering, and (d) Microwave sintering.

Relative density and porosity

Archimedes' principle and rule of mixtures [RoM] were used to calculate the relative density of the Conventional Sintered (CS) and microwave (MW) sintered samples. The relative density, sintered density and theoretical density of the CS sample and MW sintered sample is given in Table 3. The microwave (MW) sintered sample was subject to rapid heating when compared to the Conventional Sintered (CS) sample, which results in the suppression of grain growth, and an overall increase in the relative density. The relative density of the developed composites is summarized in Table 3. As discussed earlier, the nature of heating that occurs for the Microwave (MW) sintered sample led to the suppression of grain growth and the resultant high density of the composite sample when compared one-on-one with the composite sample developed using the technique of Conventional Sintering (CS). However, it is observed that the relative density of all the composites was well above 97 %. An enhancement in the relative density of the microwave (MW) sintered sample can be attributed to the conjoint and mutually interactive influences of the following: (i) nature of heating the sample, (ii) local heating of the particle-particle interface at the fine microscopic level, and (iii) role of time taken for processing. Relative density of the microwave (MW) sintered sample was found to be marginally higher than the Convention Sintered (CS) sample, as summarized in Table 1 and shown in Figure 4a. Porosity is a factor that influences density of the engineered composite material, and the following formula was used to calculate porosity of the developed composite.

Table 3: Relative density, sintered density, and theoretical density of the Conventional Sintered (CS) and Microwave (MW) sintered samples.

Sintering	Theoretical Density (g/cm^3)	Sintered Density (g/cm^3)	Relative Density (%)
CS	5.484	5.438	98.16±0.15
MW	5.484	5.445	99.29±0.10

$$Porosity(\%) = \frac{\rho_t - \rho_s}{\rho_t} \quad (1)$$

In this expression, ρt is the theoretical density and ρs is the sintered density.

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The microwave (MW) sintered sample which experienced rapid heating and higher densification led to a noticeable reduction in porosity. The porosity of the microwave (MW) sintered sample was marginally lower than that of the Conventional Sintered (CS) sample. The percentage of porosity of the CS sintered sample and MW sintered sample was found to be 0.84±0.07 % and 0.71±0.05 %. This is shown in Figure 4b.

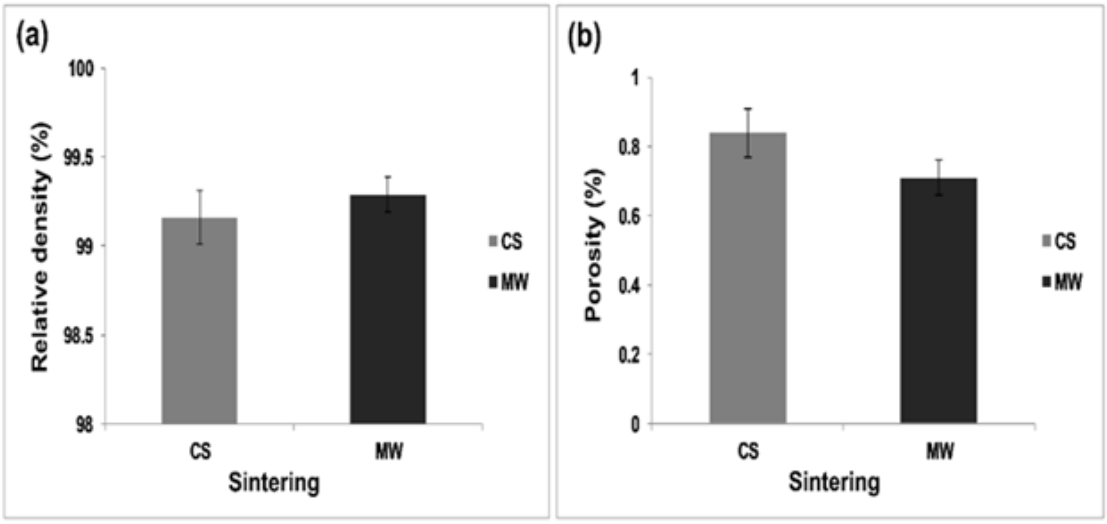

Figure 4: (a) Relative density of the CS and MW sintered sample. (b) Porosities of the CS and MW sintered sample.

Thermal conductivity

The property of thermal conductivity refers to an ability to transmit heat through a material. The ability for thermal conduction is made possible in materials due to the movement of electrons coupled with vibrations of the lattice occurring at the fine microscopic level. The hot disk sample holder and sensor thermal analyzer set up is as shown in Figure 5a. Thermal conductivity of the developed ATZ composite based on the use of alumina to reinforce the 3YSZ matrix, is shown in Figure 5b. Thermal conductivity is affected by the following factors: (i) presence of grain boundaries, (ii) internal porosity, and (iii) presence and distribution of the impurities in the ceramic sample. These factors can be carefully controlled, and the level of thermal conductivity achieved in the ceramic composite materials. The property of thermal conductivity was measured using a hot disk thermal constants analyzer on both the Conventional Sintered (CS), and Microwave (MW) sintered Alumina Toughened Zirconia (ATZ) nanocomposite samples and is shown in Figure 5a. The Microwave (MW) sintered sample was subjected to rapid heating, which led to a suppression of grain growth when compared to the sample obtained by Conventional Sintering (CS) resulting in higher densification and a smaller grain which contributed to improving the thermal conductivity of the developed composites. In Microwave (MW) sintering, the rapid heating is favorable for retaining the stability of the tetragonal zirconia phase, which results in improved thermal conductivity of the developed composite. The thermal conductivity of the Conventional Sintered (CS) sample was 2.3±0.15W/mK while thermal conductivity of the Microwave (MW) sintered sample was 2.6±0.1 W/mk. This is shown in Figure 5b.

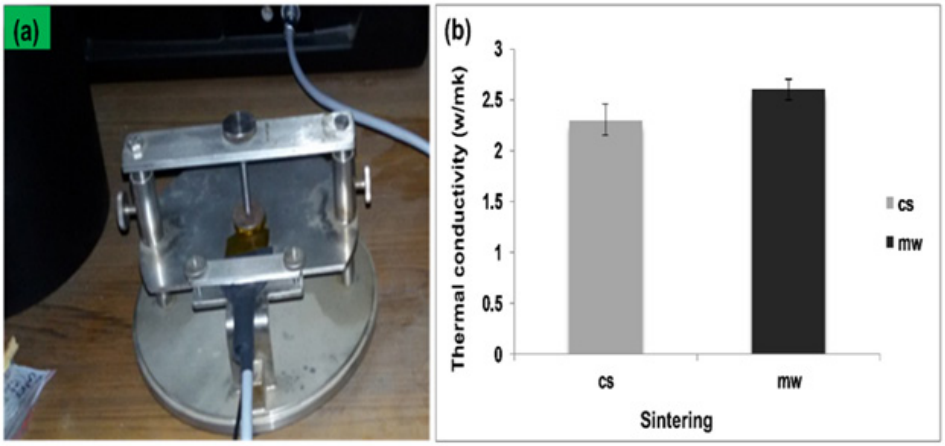

Figure 5: (a) Hot disk sample holder and sensor thermal constant analyzer. (b) Thermal conductivity of CS sintered samples and MW sintered samples.

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Conclusion

The following are a few of the key conclusions that can be drawn from this novel research study on an emerging ceramic composite material. From the X-Ray Diffraction (XRD) pattern, the stability of the t-ZrO_2 phase in the microwave (MW) sintered sample was considered higher than of Conventional Sintered (CS) sample was observed. However, it shows the highest peak intensity of the t-ZrO_2 phase and a few minor peaks of Al_2O_3 and m-ZrO_2 phase were observed. The relative density of the Microwave (MW) sintered sample is higher than the Conventional Sintered (CS) sample due to rapid heating and suppressing grain growth was observed in microwave sintering. Porosity of the Microwave (MW) sintered Alumina Toughened Zirconia (ATZ) sample is lesser than the Conventional Sintered (CS) sample due to more densification in microwave sintering. The thermal conductivity of the microwave (MW) sintered sample is higher than the Conventional Sintered (CS) sample due to rapid heat in MW sintered sample was observed the small grain size leads to improved thermal conductivity. Additional research is currently in progress to reaffirm the benefits of sintering on mechanical response and properties of the chosen and studied ceramic composure materials.

Acknowledgment

The authors would like to extend their appreciations and gratitude to the Department of Mechanical & Industrial Engineering as well as Department of Metallurgical and Materials Engineering, Indian Institute of Technology, Roorkee for providing the required facilities throughout the investigation and experiments. This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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