

Effect of microwave sintering on the hot modulus of rupture and thermal shock resistance of zirconia doped cordierite

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Cordierite ceramics are often used as kiln-furniture due to their good mechanical and Hot MOR properties. Still, there is a need to improve properties like strength at high temperatures. Cordierite ceramics were prepared using the solid-state method by doping variable amount of Zirconia particles in the recipe. The percentage of Cordierite ($2\text{MgO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$) is maintained as SiO_2 -51.4%, Al_2O_3 -34.8% and MgO -13.8%. Variable weight percentages of Zirconia particles were added to the Cordierite body and milled. The recipe is compacted and microwave sintered. Microwave furnace is used for sintering the doped Cordierite body. Particle size measurement, XRD and FESEM were carried out for all the samples. The hardness and Hot MOR tests were conducted. The physical properties of all the sintered bodies, such as bulk density, porosity and water absorption were measured by the Archimedes method. It is observed that the addition of Zirconia particles to Cordierite increased the Hot MOR of the Cordierite body. It is one good indication to recommend that the Cordierite can be used in high temperature applications with higher loads.

Key words: Cordierite, Zirconia doping, Hot MOR, Thermal shock resistance, Kiln furniture.

Introduction

The Cordierite composites are potential materials in the field of ceramics, due to their excellent physicochemical properties such as high thermal shock resistance, low thermal expansion coefficient and high refractoriness [1-6]. These mechanical properties have become evolutionary factors in the ceramic industries to improve the strengthening of the Cordierite-based products [7-9]. The development of Cordierite composite is a trustable area in the electrical and thermal applications. Cordierite has three polymorphic modifications, α -Cordierite-high temperature formation, β -Cordierite-low temperature formation and μ -Cordierite metastable in condition [10-11]. Among all the Cordierite phases, the α -Cordierites are extensively used in various applications such as electronic substrates, circuits, abrasion resistant tiles in industrial pipes and kiln-furniture applications and so on [12-13]. The growing interest towards the synthesis of Cordierite composite by the various oxides such as cerium, vanadium, bismuth, nickel, copper, zirconium and even boron has been worked by many authors [14-15]. F.A. Costa Oliveira et.al. showed a result the addition of nano ZrO_2 particle in Cordierite improved the strength and elastic moduli of the composites prepared by attrition milling. [16]. A. Hirvonen et.al. worked the yttrium stabilized Zirconia (Y-TZP) with Cordierite and produced the superior mechanical and thermal properties

of Composite [17]. En-Hai Sun et.al. revealed the formation mechanism of ZrSiO_4 in the cordierite- ZrO_2 composite in the temperature range from 1250 to 1400 °C and the phase formation initially it was observed at 1250 °C [18]. Zirconium-Cordierite ceramic composites were synthesized by a co-precipitation method using $\text{MgCl}_2\cdot 6\text{H}_2\text{O}$, NaAlO_2 , $\text{Na}_2\text{SiO}_3\cdot 5\text{H}_2\text{O}$ and $\text{ZrOCl}_2\cdot 8\text{H}_2\text{O}$ as starting materials [19]. However, Micro hardness of the sintered samples was enhanced from 7.1 to 7.5 GPa with increasing the Zr^{4+} dose from 0 to 25 wt. %. The electrical resistivity was found to be increased for the composites and the dielectric permittivity varied with the different percentage of doping of Zirconium Oxide. The Cordierite/Silicon Carbide (SiC) composite ceramics with variable wt. % Cordierite was in-situ prepared via pressure less firing technique by using silicon carbide, alumina, Kaolin and talc as basic forming materials. The results were concluding that the synthesized composites exhibited the good physical and mechanical properties and good thermal shock resistance [20]. Cordierite-mullite Composite at 70 : 30 wt% showed good mechanical and thermal properties [21]. Hence, the synthetic importance and extensive importance of the Cordierite ceramic composite materials has been raised for Kiln-furniture application. Hot MOR test gives information of Cordierite weight bearing capacity while using it as kiln-furniture. Hence more study on Hot MOR is necessitated.

In the present work, the aim is to prepare the Cordierite composite by ternary mixture ($\text{MgO} : \text{SiO}_2 : \text{Al}_2\text{O}_3$) and with different percentages of Zirconium dioxide. These studies deal with how effectively it enhances the Hot MOR properties for Cordierite composites. For getting a high

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quality of Cordierite composite formation (ZrMgSiAl), Zirconium dioxide, talc, kaolin and alumina were taken as input materials. The Cordierite formation and crystalline nature were studied by various characterization techniques in addition to the mechanical properties. The effects of Cordierite composite by microwave sintering were studied.

Experimental

Sample preparation

The materials used in this experiment were relatively pure, commercial grades of Zirconium dioxide (CDH, New Delhi), talc, kaolin, Alumina and silica (ABREF PRIVATE LTD. Tamil Nadu). The Cordierite composition was designed in such a way that it contains SiO₂-51.4%, Al₂O₃-34.8% and MgO-13.8% [22-23]. Then, additions of ZrO₂ powder in the weight proportions of 4, 8 and 12% were added to the stoichiometry of Cordierite powder. These samples are denoted as L, M and N respectively. Pure Cordierite is denoted as K. These four samples (K, L, M and N) were separately milled in an aqua medium using a tungsten carbide tank and tungsten carbide grinding media (10 mm dia.) for an optimum time period of 30 minutes for the purpose of homogeneous mixing rather than grinding. Grinding media versus the charge was maintained as 2:1. The mixing operation in a noncontaminating environment was performed using a high-energy ball mill that consisted of several batches for huge sample collection. The reason for selecting attrition process is to bring the recipe into homogeneous mixing due to variation in density of Cordierite (2.63 g/cm³) and ZrO₂ (5.40 g/cm³) [24]. The homogeneous recipes of four batches were dried at 110 °C for 24 hours. Dried samples were de-agglomerated and sieved to obtain the uniform particles as well as the uniform mixing of the powders. After sieving, the obtained powders were uni-axially pressed into bars of Length, Breadth and Height (LBH) with measurements of 150 × 25 × 25 mm for hot MOR test and pellets with a diameter of 12.5 mm for other characterization at the pressure of 250 MPa. The pressed samples were sintered in air using a microwave furnace (VB Ceramic Consultants, Chennai, India.) with the heating rate of 40 °C min⁻¹, soaked at 1275 °C for 25 min⁻¹ and then cooled to room temperature at the rate of 40 °C min⁻¹. The concept behind moving microwave furnace is in two ways benefited, one, it can be fired at the product fast and is friendly to the environment; second, less energy is enough for sintering the product [25].

Characterization

The fired samples were investigated and the crystalline phases were identified by X-ray powder diffractometer (Rigaku Mini flex IIc, Japan.) using Cu

K α (1.54) radiation in the 2 θ range of 5-70 ° at a scan rate of 5 °/min. For the preparation of a sample for this test, an agate mortar was used for making the fine powder. Obtain crystalline phases were evaluated using the reference material patterns compiled by the International Centre for Diffraction Data (ICDD) [26-27]. Based on its weight-to-volume ratio, the density and porosity of the samples were measured by the Archimedes method (ASTM 792). The microstructure studies of the samples were observed using scanning electron microscopy (FESEM-SUPRA 55-CARL ZEISS). Prior to conducting a SEM test, the sample was polished using a diamond wheel and polishing cloths for getting the smooth surface for the samples, later it was free treated with HF for 10 seconds to eliminate the loose particles on the surface. During the examination of a test, the surface charge problem was prevented by fuming of Pd-Pt alloy on the surface of samples. Particle-size distributions of all milled powders were measured by an ultrasonic particle-size analyzer (Horiba particle size analyzer, Model LA-950V2, Japan). The median size of the samples (d₅₀) for K, L, M and N were obtained as 6.33, 6.45, 7.08 and 7.16 μ m respectively. The Hardness of the samples were characterized by Vickers hardness tester (Zwick, Model- Material prufung) based on a diamond square-based pyramid and its face consists of 136 ° at the sample contact area, which gives geometrical impressions on the sample by introducing different loads ranging from 1 to 10 kg. The results were measured with the reading of average diagonal length by using the formula

$$\text{VHN} = 1.8544 \times F / a^2 \quad (1)$$

Where F = force (load) in Kg (kilograms)
a = diagonal length of the impression in mm

The Hot MOR test was conducted according to IS 1528 [28]. This test was performed on a rectangular slab with the dimensions of 150 × 25 × 25 mm. The temperature was risen to 1250 °C and held for 30 minutes at this temperature. While doing this test, the load was applied gradually at a uniform speed (0.5 mm/min.) around 5 kg/min until the failure of the specimen. Initially, the fired samples were arranged in a Hot MOR furnace and graphite elements were used as heating elements in the furnace. It is clearly shown in Fig. 1 and the rate of heating was maintained at 6 °C min⁻¹. The test procedure is similar to flexural strength (3 point bend test) but, the only difference was that the temperature was higher than the room temperature while doing the experiment. Hot MOR is a good criterion to emulate the strength of refractories for kiln-furniture applications.

The modulus of rupture was evaluated by the following formula [29]

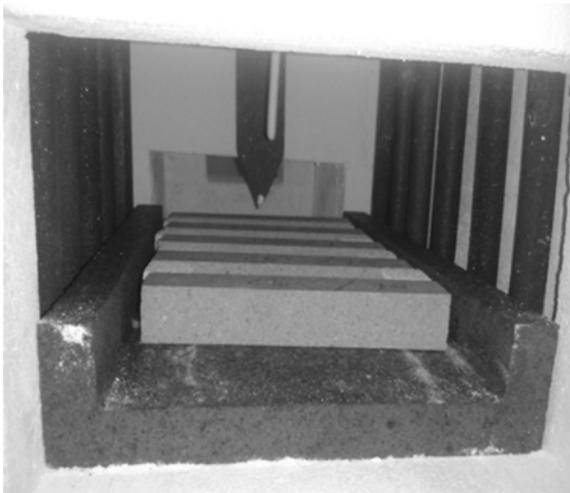


Fig. 1. Hot MOR test furnace.



Fig. 2. Thermal shock resistance test.

$$MOR = 3PL/2BD^2 \tag{2}$$

Where P is Load at rupture in Kg, L-the distance between supports in mm, B-the width of the sample and D-thickness of the sample in mm. Thermal stability of the samples was evaluated experimentally by air quench test. Fig. 2 shows the experiment process in the air quench test. Samples were cylindrical with 12.5 mm diameter and 5.0 mm thickness. The samples were transferred into a muffle furnace at 1250 °C and held for 10 min. The samples were then quenched into air and left for 10 min and returned to the furnace at 1250 °C. This procedure was repeated until the hair line crack on the surface of samples is identified. Photographs of samples were taken and the obtained cracks on the surface were identified using an optical microscope.

Results and Discussion

Chemical analysis

The raw materials for Cordierite from ABREF Pvt, Ltd, Chennai were analyzed by the gravimetric analysis method and are given in Table 1. Based on the

Table 1. Chemical analysis of the raw materials for Cordierite body.

Compound	Al ₂ O ₃ (Wt %)	SiO ₂ (Wt %)	MgO (Wt%)	Loss on ignition (Wt %)	Others (Wt %)
Talc	–	60.2	33.4	5.4	1.0
Kaolin	37.5	46.5	–	14.5	1.5
Alumina	99	–	–	0.7	0.3

stoichiometry of Cordierite compositions, these materials were adjusted that it contains SiO₂-51.4%, Al₂O₃-34.8% and MgO-13.8%. About 99.8% ZrO₂ (CDH, New Delhi) is used as dopant.

Particle size distribution (PSD) curves

The particle size was determined using the particle size analyzer (Horiba particle size analyzer, Model LA-950V2, Japan) and the results are shown in the Fig. 3. The particle size distributions of the samples of d90, d50 and d10 are presented in Table 2. The particle size distribution plays a major role on many properties like density, water absorption, porosity and abrasiveness. From the process point of view, it influences the sintering time. As the particle size is too low, it processes fast sintering, but one major problem to be concerned about is that finer particles often tend to agglomerate fast and it weakens the strength of the

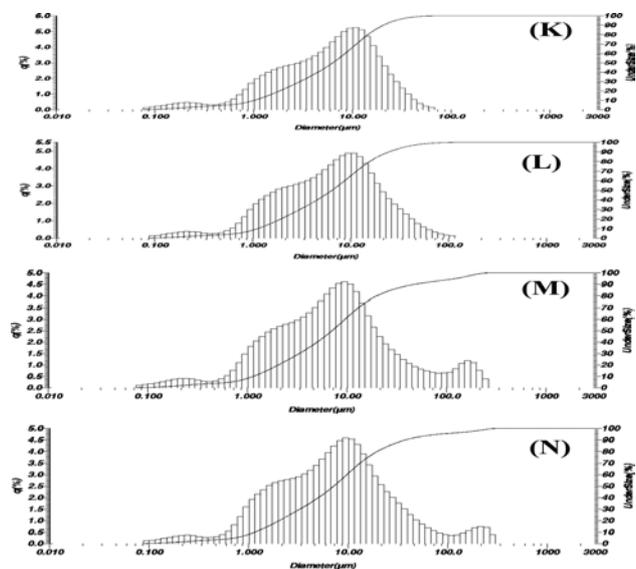


Fig. 3. Granulometric distribution curves of the samples K, L, M and N.

Table 2. Particle size distribution.

Samples	D10 (Cumulative %)	D50 (Cumulative %)	D90 (Cumulative %)
K	1.02 µm	6.33 µm	20.51 µm
L	1.12 µm	6.45 µm	24.53 µm
M	1.15 µm	7.08 µm	47.75 µm
N	1.15 µm	7.16 µm	37.49 µm

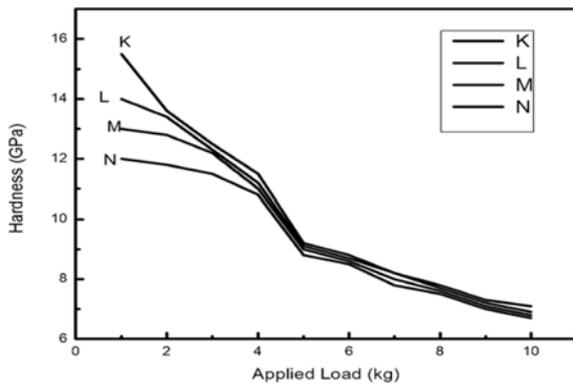


Fig. 4. Vicker's hardness of cordierite composite with variable weight load applied on K, L, M, and N samples.

Table 3. Physical properties of the cordierite specimens.

Sample	Bulk Density	Porosity	Water absorption	True density
K	2.11	22.1	11.3	2.5
L	2.34	24.3	12.4	2.6
M	2.45	25.2	13.8	2.7
N	2.61	27.1	14.5	2.9

body. [30]. Morphology of the starting samples was observed by SEM image. It depicts the even distribution of Zirconium dioxide, shown clearly in the Fig. 6. The particle size of Cordierite is finer than the Composite. It governs the improvement in hardness.

Physical properties of the Cordierite-composite

The bulk density was evaluated by Archimedes' principle and presented in Table 3. It is an indication of the degree of compaction of a specimen and calculates the theoretical weight of a specimen which is benefited for kiln-furniture applications. True specific gravity is the ratio of the density of the material to the density of the water and was tested by Pycnometer [31] using the formula.

$$\text{True specific Gravity} = \frac{(M_2 - M_1)\rho_0}{(X - Y)} \quad (3)$$

Where,

M_1 = weight of the bottle, M_2 = weight of the bottle + solid and ρ_0 = density of water

$X = M_4/M_3$, $Y = M_3/M_2$

M_3 = weight of the bottle + solid + water, M_4 = weight of the bottle + water.

The true specific gravity values were found to be 2.5, 2.6, 2.7 and 2.9 for K, L, M and N because of good crystalline formation of the Cordierite phase; it was shown in Table 3. The obtained specific gravity confirms the Cordierite body [24, 32-33]. Porosity plays a major role in the kiln-furniture application. It affects the sintering intensity, mechanical strength, thermal expansion, thermal conductivity and corrosion of products. In this experiment,

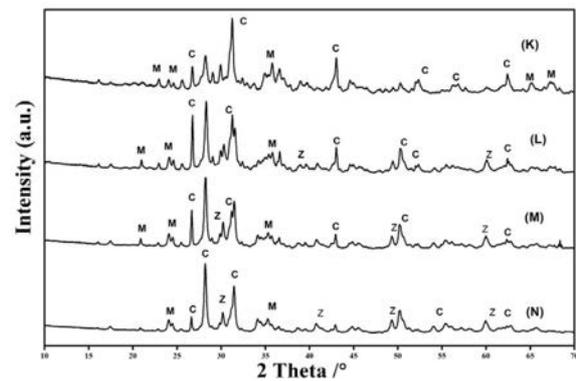


Fig. 5. The XRD patterns of the cordierite composites (k), (l), (m) and (n), the corresponding phases also indicates as Z: ZrMgSiAl A: alumina, C: cordierite, M: mullite.

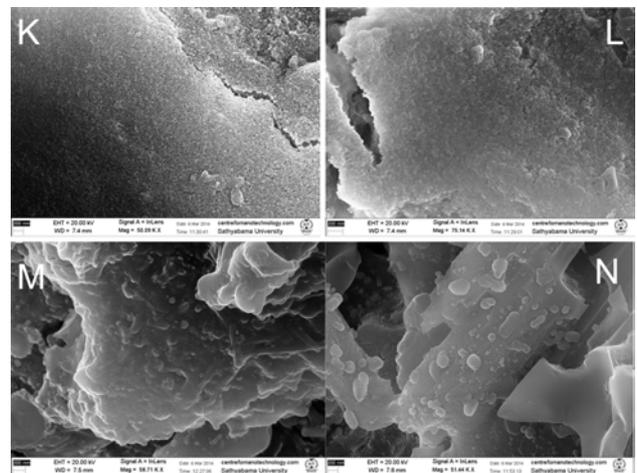


Fig. 6. Microstructures of the Cordierite composite specimens of K, L, M and N.

the obtained porosity of all samples is acceptable for cordierite body for using in kiln-furniture application. Water absorption indicates how well the samples were sintered. It was measured purely by the amount of water mass absorbed into the open pores to the mass of the specimen, whereas in porosity it includes the closed pores. It is clearly indicated in all the samples, the closed pores and open pores are more or less equally spread into the samples.

Vickers hardness

Fig. 5 shows Vicker's hardness for the variable weight load applied on Cordierite-Zirconia composite samples. The Vicker's hardness of K specimen is 15.5 GPa for an applied load of 1 kg which decreases to 7.1 GPa for 10 kg of applied load. However, the N sample is 12.0 GPa for an applied load of 1 kg and decreases to 6.7 GPa for 10 kg. The Vickers hardness value of the pure Cordierite is comparable to the pure Cordierite by Toshio Ogiwara et al. [34]. The hardness value is observed for K sample little higher than the composite bodies due to the formation of α -Cordierite, spinel phase may be the reason to achieve these values.

One more indication, the grain growth was not observed for the samples K as revealed in Fig. 6.

Hot MOR studies on Cordierite-composite

The Hot MOR test can be used to indicate the loading capacity and resistance to the deformation of kiln-furniture. The test process is simple [35], it measures the maximum transverse breaking stress applied that a material can withstand before fracture at an elevated temperature. In two ways this test is useful, one is the quality of a product is maintained to ensure the consistency between different batches used in the working process, and another is to estimate the loading capacity, deformation of products, thickness and lifetime of Cordierite products while it is used in kiln-furniture applications. Based on Formula 2, the Hot MOR for Cordierite specimens was found to be 92, 102, 112 and 120 kg/cm² for K, L, M and N respectively. Test process was revealed in Fig. 1. The Hot MOR results were achieved higher even though the particle size of N sample is little higher than the specimen of K. From the theory point of view, the bending strength often depends on particle size and its distribution; phase formation; pore size and its distribution and micro-structural uniformity. Probably the L, M and N samples consist the zirconium oxide which reacts with SiO₂ to form Zircon around 1200 °C and above. Based on these results, it may come to conclude that the zirconium dioxide doped Cordierite body can be used as kiln-furniture at high load capacity.

Phase composition of Cordierite-composite

The X-Ray diffraction patterns of the Cordierite and Cordierite-zirconia composites are shown in Fig. 5. The XRD data are given in Fig. 5, shows that Cordierite material is containing minor proportions of zircon composites (ZrMgSiAl) in L, M specimens and it appears more clearly in N specimen. XRD spectra indicate that the formation of zircon (ZrSiO₄) phase with a little amount of a glassy phase in the tannery mixtures may be a probable reason to improve the Hot MOR strength. It was revealed in SEM image also (Fig. 6). The Zircon phase of composite 2 theta values at 26.6°, 35.4°, 68.3° for specimen N and M are matched with the ICDD files (JCPDS NO. 83-1374, 89-2655 and 75-1590, 75-1564). All other obtained peaks were confirmed based on the ICDD numbers for Cordierite (JCPDS No: 89-1487: 48-1600), and mullite (JCPDS No: 79-1457: 15-0756). Cordierites reacted with ZrO₂ particles and continuously improve the Zircon phase as shown in Fig. 6. It is one good evidence that higher zircon phase was gathered by the high percentage of ZrO₂ addition. It was depicted on elsewhere (8, 36).

Microstructure of cordierite-composite

Micrograph of the Cordierite composite specimen, obtained by sintering at 1275 °C is presented in Fig. 6.

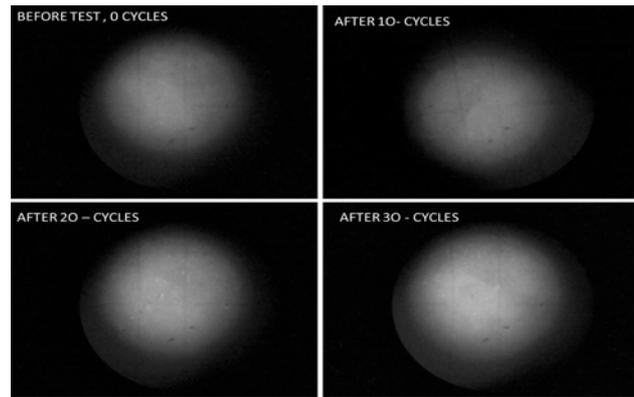


Fig. 7. Photo image of K sample.

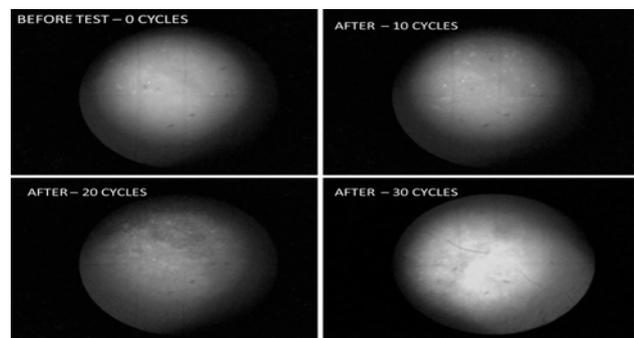


Fig. 8. Photo image of N sample.

Distribution of zirconia through the composite appeared homogeneous in all the M and N specimens, this homogeneity could be enhancing the mechanical properties of the samples. The phase of zircon (ZrSiO₄) was observed in N sample as it contained around 12 wt% ZrO₂ as a dopant, already revealed by XRD (Fig. 5). From N image, Hot MOR test indicates more amount of a needle-like mullite structure which causes excellent bending resistance. The presence of ZrSiO₄ in Cordierite shows a glassy phase in the vicinity of grain boundaries as revealed in Fig. 6 and particularly in the N sample. By the assistance of residual glassy phase, the ZrO₂ and SiO₂ can be reacting around 1200 °C and form as Zircon (ZrSiO₄). It is one good indication to use the Cordierite products as kiln-furniture with the high load at a higher temperature.

Thermal shock resistance of the Cordierite-composite

Thermal shock resistance is a major property for kiln-furniture applications. It measures the ability of a specimen that has to resist the stress or breakage caused by rapid changes in temperature. Mostly, it was concerned about the lifespan of kiln-furniture applications. Two of those samples were tested and demonstrated the results in Figs. 7 and 8 for the samples of K and N respectively. For sample K, every 10 cycles the image was observed, it was indicated no sign of cracks even when it was run up to 30 cycles. For sample N, hair line cracks were observed after 30 cycles. In general, if the material has

lower thermal expansion coefficient and higher thermal conductivity, they have better thermal shock resistance. However, it is stated by many authors that the zirconia coefficient of expansion ($8.0 \times 10^{-6} / ^\circ\text{C}$) and Cordierite coefficient of expansion ($2.0 \times 10^{-6} / ^\circ\text{C}$ to $4.0 \times 10^{-6} / ^\circ\text{C}$) are varied [16, 34]. Once the temperature changes rapidly, the deformation will be constrained and a stress is generated, this thermal stress leads to thermal shock and causes the specimens get cracked, spalling or even fracture. This may be the cause that the 12% zirconia in the Cordierite may be varied with the thermal expansion and gets hairline crack at around 30 cycles. It is one evidence that the lifespan of doped zirconia Cordierite-composite is shorter than the pure Cordierite.

Conclusions

Cordierite-Zirconia composites were prepared by the solid-state method, from a ternary mixture of the $2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$, and zirconium dioxide. The preparation was achieved by the various amount of zirconia precursor doped to the talc-clay-alumina mixture to prepare the pellets and followed by drying and microwave sintering. Vickers hardness of cordierite composite specimens is little lower than those of pure cordierite due to a bit formation of agglomerate in the composite. Cordierite was sintered in this experiment at 1275°C by using microwave sintering than that of the conventional process which is fired at 1350°C . By doping in cordierite with zirconium oxide, high hot MOR was found. These results show two-way benefits for ceramic industries, one is saving energy by reduction of temperature and use the Cordierite product with more load as kiln furniture in ceramic industries, so it can produce more tons of products in short time. Hence, the studies on mechanical and thermal properties confirm that the developed Cordierite composite is suitable for high-temperature kiln furniture applications with high load.

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