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ZIRCONIA'S CHARACTERISTIC INFLUENCE ON CORDIERITE MECHANICAL PROPERTIES

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The pure cordierite and cordierite-zirconia (5-20%) ceramics were prepared by using precipitation technique. For the prepared samples, the mechanical properties such as hardness, fracture toughness, flexural strength and thermal expansion behavior were analyzed. Vickers hardness of the cordierite-zirconia samples increased with increasing sintering temperatures than pure cordierite samples. It has maximum hardness value at 1100 ºC and above this temperature; the hardness reduces with increasing sintering temperatures. The addition of zirconia (5 to 20 wt %) with cordierite increased the fracture toughness from 2.73 to 3.81 MPa. The flexural strength of composites increased from 117.28 to 278.39 MPa. The thermal expansion coefficient of cordierite and cordierite-zirconia showed an increase in magnitude from 4.08 X 10^{-6} /K to 4.89 X 10^{-6} /K. It shows the detrimental effect due to the addition of zirconia.

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1. Introduction

Cordierite ceramic has been grown into prominence in the international market because of its superior properties like low pressure drop, chemical inertness, fast heat up time and structural stability at high temperatures, low-operating costs. This proved to be a potential player in variety of application such as automotive, electronics as well as micro-electronic components, structural, molten metal filter, industrial heat recovery, water filtration, etc. [1-5]. However, pure cordierite has poor mechanical properties and very narrow sintering range, this needs to be improved by developing better methods to synthesize cordierite. Also there has been extensive research is in progress to simultaneously improve the mechanical property, thermal stability of cordierite material new materials as a dopant such as zirconia, yttria, ceria, lanthanum oxide, titanium oxide [2, 3].

However, the present research work is focused on to synthesis cordierite and cordierite-zirconia composite using a simple approach of precipitation technique with high homogeneity. For the synthesized material the mechanical properties were examined in comparing with pure cordierite and cordierite-zirconia (5-20 wt%) composite by varying compositions and to find its suitability for use as exhaust applications in automotive and industries applications. The dried and pressed green body samples were sintered at various temperatures from 600 to 1400 ºC for 3h. For the sintered samples various mechanical properties were studied to evaluate the hardness, fracture toughness, flexural strength and coefficient of thermal expansion.

2. Experimental procedure

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Cordierite and cordierite-zirconia composite was prepared by using precipitation technique. Initially raw powders of the starting materials (MgO: 13.8, Al_2O_3: 34.8 and SiO_2: 51.4) were mixed thoroughly with stoichiometric compositions to form pure cordierite (CZR0) and cordierite-zirconia composite (CZR5, CZR10, CZR15 and CZR20). Using distilled water as a solvent with continuous stirring, impregnating the samples by adding carboxy methyl-cellulose (1 wt%) as a binding agent, ethylene glycol (1 wt%) was added as a flux to facilitate easy shaping, sodium hydroxide (1 wt%) [11] is used as a lubricant and sodium carbonate (1 wt%) as precipitating agent. The mixtures of these compounds were stirred continuously for 5h to obtain high homogeneity in the precipitate slurry. The preformed cordierite samples were oven dried at 100 °C for 12h. The dried powder was again ball milled for 5h. The precursor powder (CZR0, CZR5, CZR10, CZR15 and CZR20) were then compacted in the form of pellets at 240 MPa [7] to 10 mm in diameter. Further, the compact samples sintered at various temperatures from 600 to 1400 °C for 3h .The sintered samples were then polished using different grades of polishing sheets and suitably etched for 30 minutes. The micro hardness measurements were carried out on the etched specimens using Vickers indentation method for different small loads using zwick hardness tester followed by fracture toughness by indentation-fracture method and flexural strength measurements using three point bending experiment in Unitek universal testing machine. The microstructures of sintered specimen were examined by SEM, JEOL-JSM. The thermal expansion coefficient was measured using thermo mechanical analysis in dilatometer; model VBCC from 70 to 800 ºC at a heating rate of 10 ºC/min.

3. Results and discussions

3.1 Vickers Hardness

Fig. 1 shows Vickers hardness for the various sintering temperatures of cordierite and cordierite-zirconia composite samples. The Vickers hardness of pure cordierite (CZR0) is 25.42 GPa for an applied load of 0.5 kg and decreases to 8.65 GPa [12] for 10 kg of applied load for the sample sintered at 1100 °C. The high hardness value is due to the formation of α-cordierite, spinel phase and discrete grain growth [10]. The hardness value is higher at this temperature and decreases with increase of sintering temperatures. The Vickers hardness value of the pure cordierite decreases with increase in applied load for particular sintering temperature shown in figure 2. Also the hardness value of the pure cordierite obtained at 1400 °C for an applied load of 10 kg is 7.18 GPa, which is slightly less than the standard value [13]. Figure 3 shows the maximum hardness value of 7.18 GPa for the applied load of 10 Kg for the zirconia doped cordierite (CZR20) at 1400 °C and the hardness value increases to 9.84 GPa with increase in sintering temperature for the applied load of 0.5 kg given in figure 4. The increase in hardness value for increasing sintering temperature is due to the addition of zirconia. The Vickers hardness value at 1100 °C for the cordierite-zirconia composite is decreased compared to that of pure cordierite material is mainly due to the formation of spinel as a secondary phase.

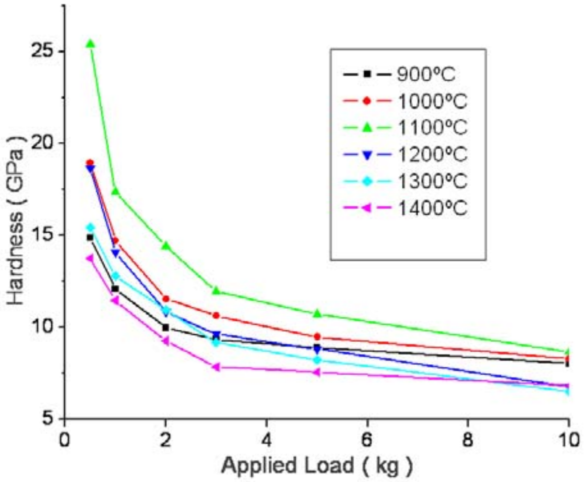


Fig.1. Vicker's Hardness of Cordierite for Various Applied Loads Sintered at Different Temperatures

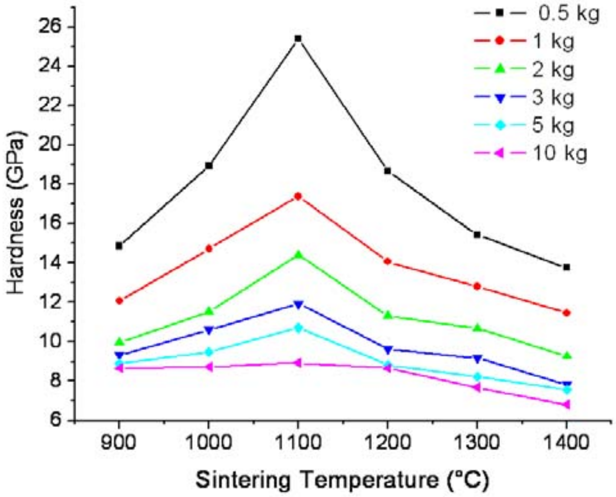


Fig. 2. Vicker's Hardness of Cordierite Sintered at Different Temperatures for Various Applied Loads

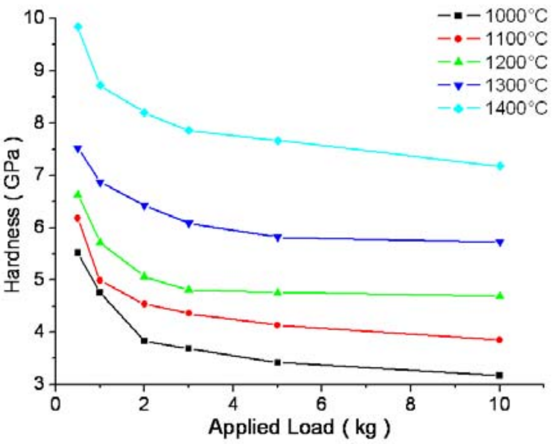


Fig. 3. Vicker's Hardness of Cordierite-Ziconia (20 wt %) at Various Applied Loads Sintered at Different Temperatures

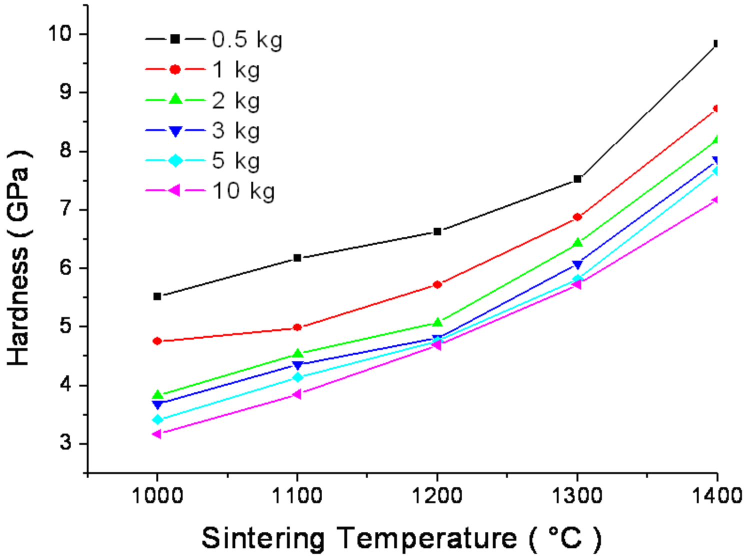
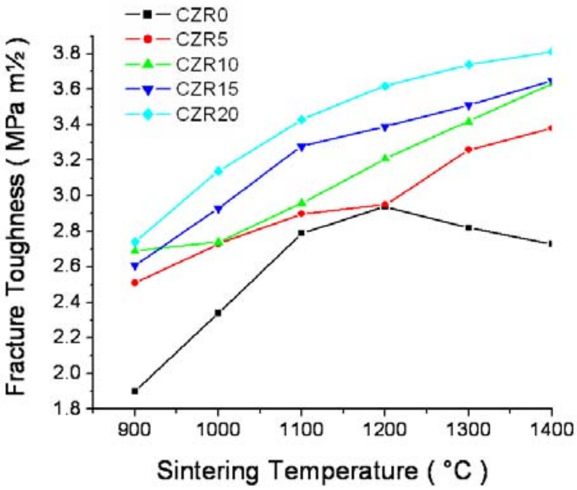


Fig. 4. Vicker's Hardness of Cordierite-Zirconia (20 wt %) Sintered at Different Temperatures for Various Applied Loads

3.2 Fracture Toughness

The fracture toughness of pure cordierite (CZR0) and cordierite-zirconia (CZR5, CZR10, CZR15 and CZR20) composites for the samples heat treated at various sintering temperatures 900, 1000, 1100, 1200, 1300 and 1400 °C for 3h respectively. The data compiled reveals that the fracture toughness of compacts depends upon the sintering temperature and crystalline phase formed in the system**.** Figure 5 shows the fracture toughness of pure cordierite material heat treated at different temperatures. The fracture toughness of pure cordierite material is low at 900 ºC. Above 1000 ºC, there is an increase in fracture toughness value and is about 2.94 MPa m^{½}.This may be due to the formation of α-cordierite above 1000 ºC. Due to the higher grain growth, this results in lowering fracture toughness with further increase in sintering temperatures [5].



Figt. 5. Fracture Toughness of Cordierite and Cordierite-Zirconia (5-20 wt %) as a Function of Sintering Temperature

Fig. 6. Optical Micrographs of typical Indentation Marks on CZR 20 wt% sample sintered at 1400 °C

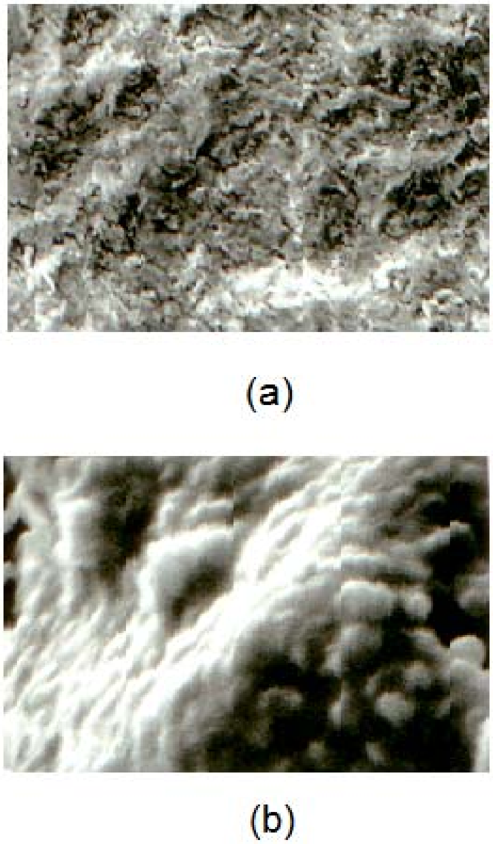


Fig. 7. SEM Photographs of Fracture Surface of Specimens sintered at 1400 °C for 3h (a) Cordierite and (b) CZR 20 wt%

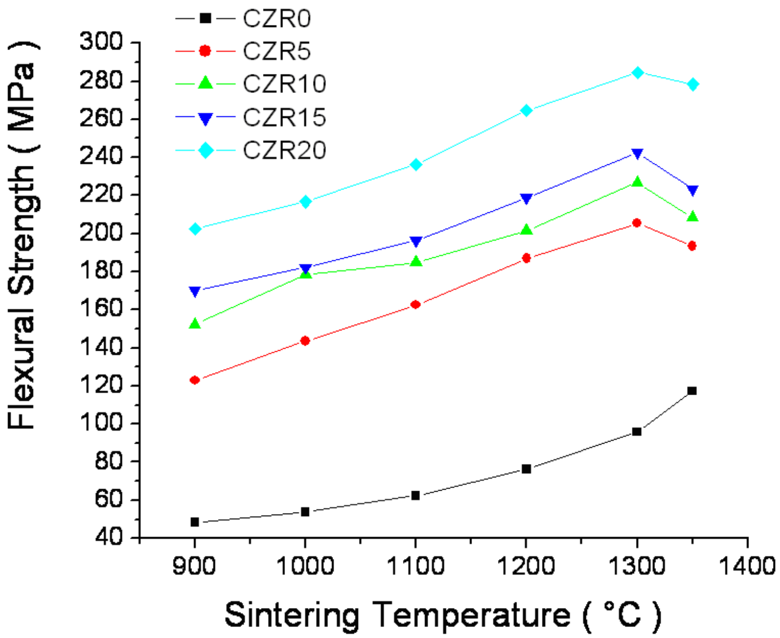


Fig. 8. Flexural Strength of Cordierite and Cordierite-Zirconia (5-20 wt %) Sintered at Different Temperatures

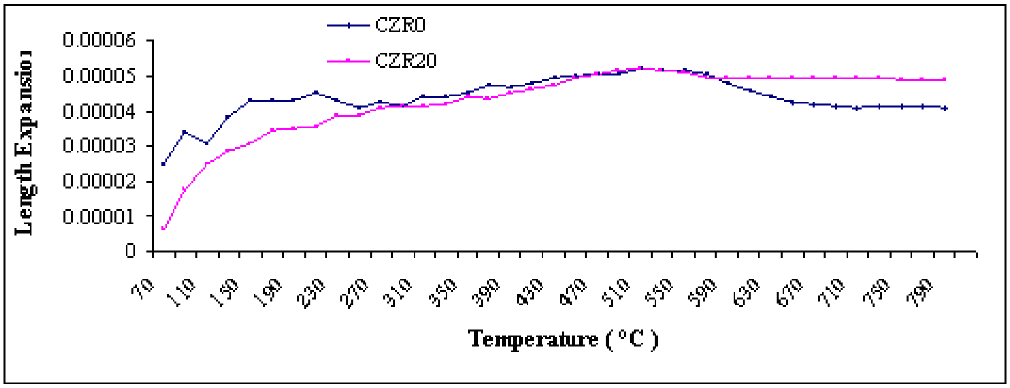


Fig. 9. Thermal Expansion Coefficient of Cordierite and Cordierite-Zirconia Sintered at 1400 °C

The fracture toughness of the cordierite is also enhanced with the addition of zirconia .Generally; toughening of ceramics with zirconia can be achieved through martensitic transformation from tetragonal to monoclinic phase with zirconia and it exhibits a maximum value 3.81 MPa m^{½}[8] at 1400 ºC for 3h which can be measured from crack length of indented specimen (Figure.6). The fracture toughness value of cordierite-zirconia composite increases as the sintering temperature increases with the addition of zirconia (5-20 wt%) compositions upto a maximum sintering temperature of 1400 ºC. This may be due to decrease in t-Zirconia phases. The zirconia reacts with cordierite to from reaction products of cordierite, zircon and m-zirconia [14]. The toughening may be either due to stress induced transformation of particles near to the crack zone or predominantly because of the presence of spinel, zircon and m-zirconia particles that exhibits higher toughness values. The fracture toughness value of composites is higher for the addition of 20 wt% of zirconia than 15wt% of zirconia. Scanning electron micrograph of fracture surfaces of pure cordierite and cordierite-zirconia composite specimens sintered at 1400 ºC are shown in figure 7(a,b). Cordierite specimens reveal fine micro-pores (Figure 7(a)) whereas cordierite containing 20 wt% of zirconia (Figure 7(b)) attains a pore free microstructure.

3.3 Flexural Strength

The flexural strength of pure cordierite (CZR0) and cordierite-zirconia (CZR5, CZR10, CZR15 and CZR20) composites are obtained for the samples heat treated at various sintering temperatures 900, 1000, 1100, 1200, 1300 and 1400 °C respectively for 3h. The results shown in figure 8 reveal that the flexural strength of cordierite increases from 48.38 MPa [900ºC] to 117.2 MPa [1400 ºC] by increasing the sintering temperature, this is due to formation of α-cordierite at these temperatures. The flexural strength of cordierite-zirconia increases with increase in sintering temperature upto 1300 ºC and then slightly decreases due to formation of excess glass phase along the grain boundaries. The flexural strength of the sample CZR20 increased with increase in sintering temperatures upto 1300 °C from 202.56 MPa to 284.65 MPa. The studies show that with increase in addition of zirconia wt% the flexural strength increases and higher in comparison with pure cordierite.

3.4 Thermal Expansion

The thermal expansion for pure cordierite sample (CZR0) and cordierite-zirconia sample (CZR20) at sintering temperature 1400 ºC was performed using dilatometer between temperature range 70 to 800 ºC is shown in figure 9. The thermal expansion value for both samples is nearly linear. The results for the cordierite samples shows that the thermal expansion coefficient is about 2.48 x 10^{-6}/ºC at low temperature, and it slightly increases to 4.08 x 10^{-6}/ ºC [8] at higher temperature in comparison with works the results have been confirmed with previous results. The results shows that for the cordierite-zirconia (CZR0) samples, it shows the expansion curve increase in thermal expansion coefficient from 0.65 x 10^{-6}/ºC to 4.89 x 10^{-6}/ºC [5] respectively for the same temperature range. Even though the thermal expansion coefficient of cordierite-zirconia (CZR20) is slightly higher than the pure cordierite is basically due to the dispersion of zirconia particles. The basic difference in the curve is principally due to chemistry rather than structure, since the atomic arrangement depends on fabrication conditions and composition. The principal reason for the difference in expansion curve of both samples is because of the presence of particles like zircon, α-alumina, cristobalite with large thermal expansion coefficient for cordierite-zirconia (CZR20). However, it is clear that the zirconia has a higher coefficient of expansion (8.0 X 10^{-6} /°C) compared to pure cordierite (2.0 X 10^{-6}/ºC to 4.0 X 10^{-6}/ºC). The presence of these particles influence the cordierite-zirconia, causing a thermal expansion strain [8] also the thermal expansion mismatch between cordierite and zirconia is responsible for generation of micro-cracks.

4. Conclusion

The Results derived from the mechanical studies on pure cordierite and cordierite-zirconia (5-20 wt%) are as follows:

- The hardness value of the pure cordierite sample is higher than cordierite-zirconia composite.
- The fracture toughness values of cordierite at 20 wt% of zirconia possess the high value of 3.81 MPa $m^{1/2}$ in comparison with pure cordierite value 2.878 MPa, which results due to the presence of zircon, spinel and m-zirconia phases.
- The flexural strength increased from 117.28 MPa to 283.7 MPa on dispersion of zirconia particles.
- The addition of zirconia shows a detrimental effect on thermal expansion coefficient of cordierite.
- Hence the studies on mechanical properties confirm the cordierite-zirconia's suitability in exhaust applications.

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