

## Effect of fly ash on the development of ZrO<sub>2</sub>- Cordierite composite

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### Abstract

Synthesis of cordierite (5SiO<sub>2</sub>.2MgO.2Al<sub>2</sub>O<sub>3</sub>) has attracted special attention from the researchers for its special characteristics. Solid state sintering is the most common method of cordierite preparation using kaolinite clay, talc, alumina and fly ash with the addition of 5% zircon. The powdered samples were thoroughly mixed and compacted in the form of rectangular bar by uniaxial pressing and sintered at temperatures ranging from 1100 to 1300°C for 1 hour soaking. The amount of densification of the sintered specimens were examined by linear shrinkage, apparent porosity, true density and flexural strength measurements and also by thermal shock resistance, coefficient of thermal expansion, microstructural analysis and XRD analysis. It was observed that up to 15% may be added to the composition to produce ZrO<sub>2</sub>-Cordierite ceramics with good thermomechanical properties compared to the industrial cordierite.

**Key words:** Cordierite, talc, kaolin, fly ash, sintering, microstructure.

### Introduction

Fly ash is a waste material of thermal power station. It is a fine, glass powder recovered from the gases of burning coal during the production of electricity. These micron-sized earth elements consist primarily of silica, alumina and iron. India at present produces around 175 Million MT of ash per annum. The power requirement of the country is rapidly increasing with increase in growth of the industrial sectors. India depends on thermal power as its main source (around 80% of power produced is thermal power); as a result the quantity of ash produced shall also increase. Indian coal on an average has 35% ash and this is one of the prime factors which shall lead to increase ash production and hence, its disposal/effective utilization is one of the major problems of the day. The utilization of different types of industrial wastes for various types of tailor made products is being attempted throughout the world. There has been a growing consciousness prevailing throughout the country regarding the safe disposal and utilizing for gainful purpose to solve environmental pollution. If

we consider the global scenario, attempts have been made to produce structural ceramic products such as building bricks, tiles, cement, etc. with these materials.

The mineral cordierite is of great importance in commercial field because of extremely low coefficient of thermal expansion and thermal shock resistance. Cordierite ceramics can be synthesized by various methods [1-5]. For enhancing the reactivity of the ingredients, sol-gel process has been used to generate cordierite phase by controlled crystallization from glassy phase [6-8]. The aim of the proposed project is to use an important industrial waste material such as fly ash for the development of ZrO<sub>2</sub>-Cordierite composites.

### Experimental

The raw materials used in the present investigation were fly ash (Bakreswar Thermal power plant, Birbhum), China clay (Birbhum), Talc (Rajasthan), laboratory grade active Alumina (EMerck) and Zircon (Bihar). The china clay was received in lump form while fly ash, talc, alumina, and zircon were received in powder form.

All the raw materials were characterized properly. Four different body compositions were prepared as per the following table and then 5% Zircon was added to each batch. Milling was carried out in a pot mill. The body slip was dried in a hot air oven at 110°C.

Batch	Constituents (%)			
	Talc	China clay	Fly ash	Alumina
B I	50	40	0	10
B II	45	35	10	10
B III	45	30	15	10
B IV	40	30	20	10

Table-I: Batch compositions

Before fabrication 90% of the fine powder of each batch was calcined at 900°C and mixed with

remaining 10% uncalcined powder which acted as green bond. Then the powders were compacted in the form of rectangular bars using uniaxial pressure of 250 kg/cm<sup>2</sup>. After proper drying the pressed fabricated shapes at 120°C they were fired at different temperatures ranges from 1100°C to 1300°C for a fixed soaking period of one hour.

**Result & Discussion**

The chemical compositions of all the starting raw materials was done by X-ray Fluorescent spectroscopy and are given in Table-II.

Constituents (wt %)	Raw materials			
	Fly ash	China clay	Talc	Zircon
SiO <sub>2</sub>	58.97	44.45	61.56	35.49
Al <sub>2</sub> O <sub>3</sub>	19.23	36.93	2.50	--
Fe <sub>2</sub> O <sub>3</sub>	2.81	2.54	0.02	--
CaO	4.30	--	0.49	--
MgO	8.95	0.57	30.94	--
ZrO <sub>2</sub>	--	--	--	64.17
LOI	0.84	14.08	4.59	--

Table II: Chemical analysis of the raw materials

Physico-chemical properties of fly ash are determined by using the standard method and are given in following table

Properties	Results
Particle size	0.5-27.3 μm
Surface area	75 m <sup>2</sup> /g
Specific gravity	2.11
Carbon content	0.76 %

Table III: Physico-chemical properties of fly ash

X-ray diffraction analysis of Fly ash shows the presence of Mullite as the major crystalline phase along with small amount of quartz, hematite and magnesite. Magnitude of densification was examined by measuring the properties like linear shrinkage, apparent porosity, true density, flexural strength, and thermal shock resistance. Coefficient of thermal expansion, microstructure, XRD were also examined.

A simple general and efficient method was used to prepared the ZrO<sub>2</sub>-Cordierite composite bodies. The first change of texture in relation to sintering was shrinkage which was related to particle

agglomeration, liquid formation and subsequent pulling the grains. The phenomenon of secondary crystallization also contributed shrinkage. The nature of the shrinkage-temperature curve (fig. 1) remained same at all the batch compositions. Variation of shrinkage is not of high order; it is from about 11 to 14% and directly related to fly ash content. Initial shrinkage might be due to the liquid formation followed by crystallization.

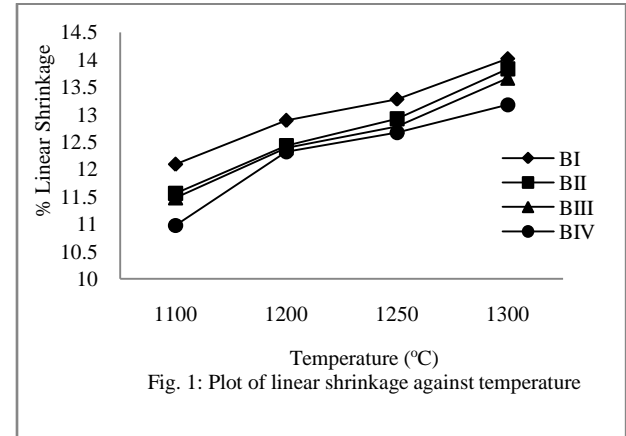


Fig. 1: Plot of linear shrinkage against temperature

Before firing the entire porosity is present as open pores. During firing the volume fraction of porosity decreases (fig. 2). As the sintering proceeded through the decrease in free energy particle migration occurred through the formation of thermodynamically more stable phase which was cordierite in this case. The general trend was porosity decreased with temperature. The low porosity values indicate that densification increased with cordierite formation.

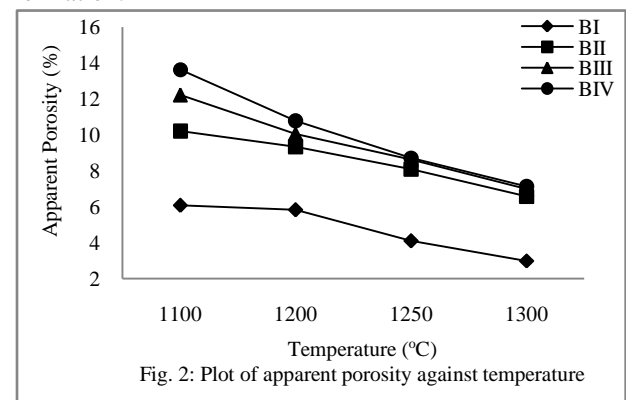


Fig. 2: Plot of apparent porosity against temperature

Change of true density in a multiphase system is an indication of phase transformation occurring with the change of temperature. True density (fig. 3) followed an inverse relationship with fly ash content which might be due to lesser amount of liquid formation.

The density values of the fired samples are found to be in the range of 2.35 to 2.71 gm/cc.

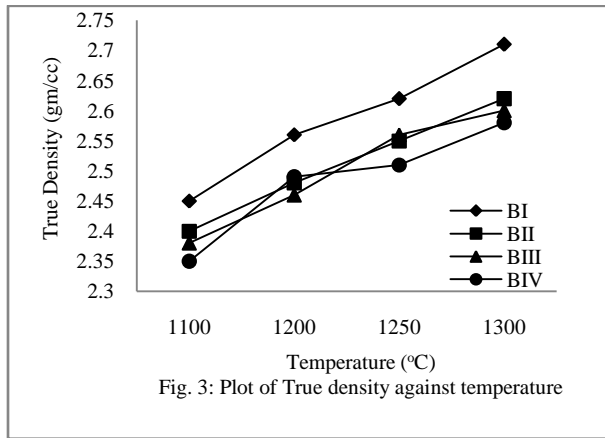


Fig. 3: Plot of True density against temperature

The mechanical properties of a polycrystalline material are very much dependent on the inherent bond nature and also on the formed microstructure. The flexural strength (Fig.4) is significantly high and in each case maximum strength is achieved at 1300°C

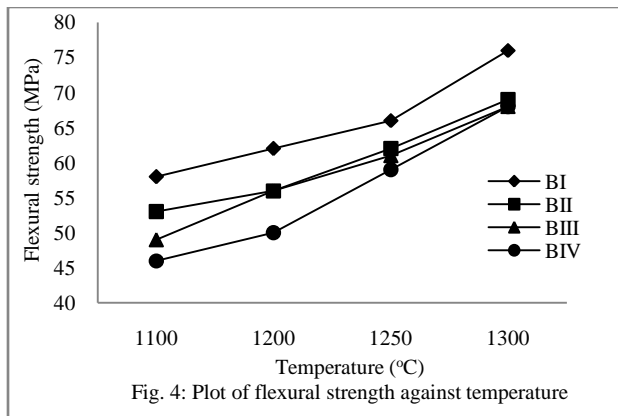


Fig. 4: Plot of flexural strength against temperature

After offering the thermal shock there is reduction in strength but that is not of high order. Co-efficient of thermal expansion of the experimental samples are in the range of  $2.9 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$  to  $3.6 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$  which are near to the industrial cordierite.

XRD analysis was carried out for characterizing the developed crystalline phases and are shown in fig. 5-8. X- ray diffraction pattern of the samples fired at 1300°C indicates the presence of cordierite as the desired phase along with small amount of mullite and t-ZrO<sub>2</sub> in the matrix.

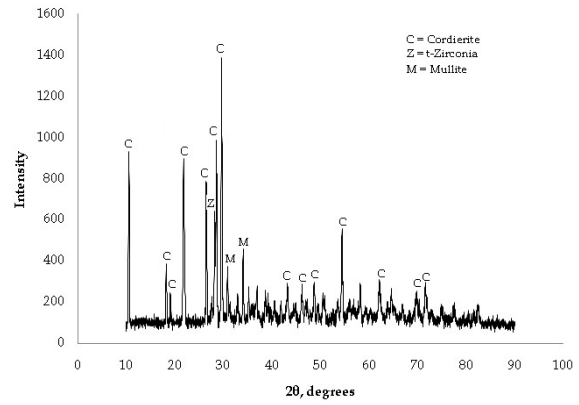


Fig. 5: XRD pattern of BI fired at 1300°C

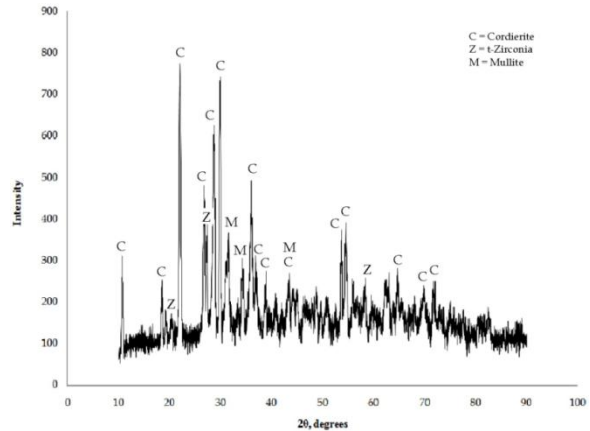


Fig. 6: XRD pattern of BII fired at 1300°C

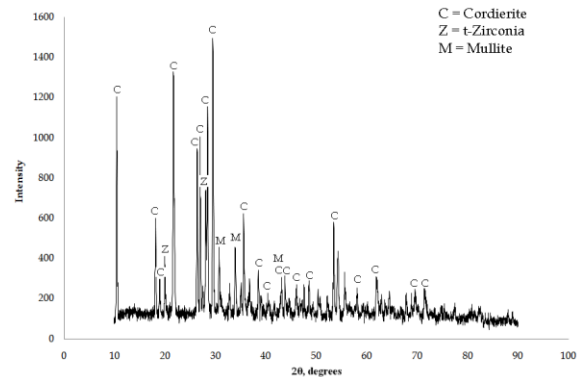


Fig. 7: XRD pattern of BIII fired at 1300°C

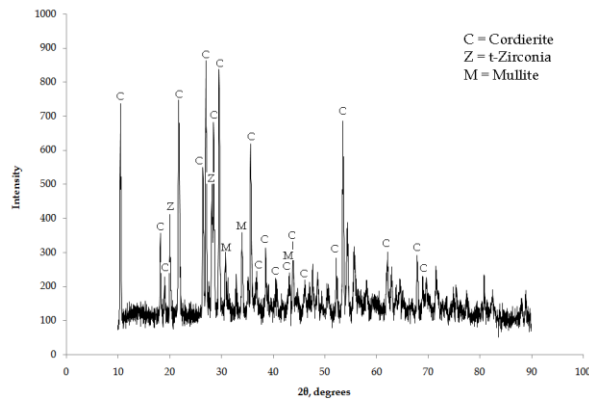


Fig. 8: XRD pattern of BIV fired at 1300°C

The assessment of the phase assemblage in the microstructure was studied through scanning electron microscopy of the fractured surface of the samples fired at 1300°C (fig. 9a-d). In all the microstructure homogeneous nature of the product was observed with sub-rounded grains.

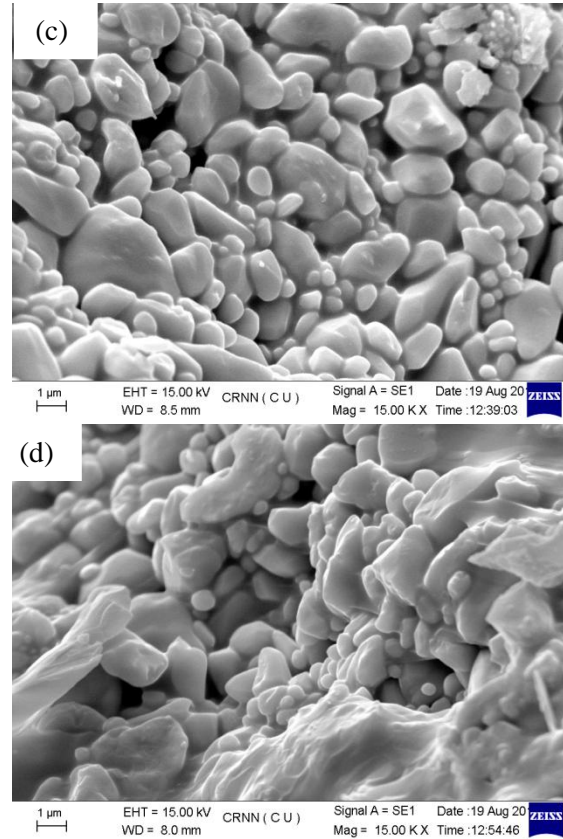
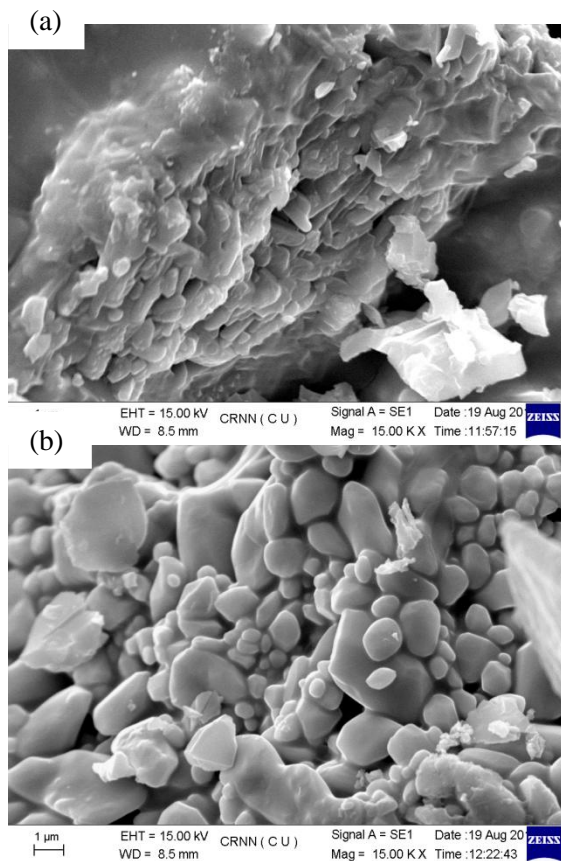


Fig. 9: Scanning Electron Micrograph of samples fired at 1300°C, [(a)-BI, (b)-BII, (c)- BIII, (d)- BIV]

### Conclusion

Cordierite-ZrO<sub>2</sub> body can successfully prepared by using about 15% fly ash. Its low linear thermal expansion coefficient and high compressive strength is near to the industrial cordierite products like cordierite honeycombs, cordierite kiln furniture, etc. In addition, the utilization of fly ash in the Cordierite-ZrO<sub>2</sub> body can effectively reduce the negative impact of this solid industrial waste on the environment; considerably decrease the production costs of relative industrial products, and efficiently save limited natural resources.

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## **Author Profile**



Dr. Arabinda Mondal obtained the B. Tech Degree in Chemical Technology (Specialisation in Ceramic Engineering) from University of Calcutta in 1999 and M. Tech Degree in Ceramic Engineering from the same University in 2001. He joined as a lecturer in Ceramic at the Department of Silpa-Sadana (Pottery-Ceramic Division) of Visva-Bharati University in the year 2001. Presently he is working as the Associate Professor in the same department and also as the Vice-Principal of Palli Samgathana Vibhaga, Visva-Bharati. He received his Ph.D. Degree from the University of Calcutta in the year 2005. Dr. Mondal has done lots of work in fine ceramics, fly ash utilisation and ceramic composites. He has published several papers in different national and international journals.