SYNTHESIS AND CHARACTERIZATION OF CORDIERITE-MULLITE COMPOSITE

A THESIS IN THE PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

BY

ANKITA BILUNG

ROLL NO: 108CR016



DEPARTMENT OF CERAMIC ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA

SYNTHESIS AND CHARACTERIZATION OF CORDIERITE-MULLITE COMPOSITE

A THESIS IN THE PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

BY

ANKITA BILUNG

ROLL NO: 108CR016

Under the guidance of

Prof. SWADESH KUMAR PRATIHAR



DEPARTMENT OF CERAMIC ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA



NATIONAL INSTITUTE OF TECHNOLOGY, ROURKELA

2008-2012

CERTIFICATE

This is to certify that the thesis entitled, "*Synthesis and Characterization of Cordierite and Mullite composite*" submitted by Miss. ANKITA BILUNG in partial fulfillment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by her under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree or Diploma.

Prof. S.K.PRATIHAR Dept. of Ceramic Engineering National Institute of Technology Rourkela – 769008

Date: 10th May, 2012

ACKNOWLEDGEMENT

This project report could not have been prepared, if not for the guidance, help and encouragement from Ceramic Department, NIT Rourkela.

I take pleasure in thanking my project guide Prof. S.K.Pratihar, Department of Ceramic Engineering for introducing the present topic and for his valuable suggestion, constructive criticism and inspiring guidance throughout this project work. I would also like to express my gratitude to Prof. J. Bera (Head of the Department), and all the professors for their valuable suggestions and encouragements at various stages of the work.

I am thankful to all staff members and research scholars who have always been there to guide and help their way best in this project. And also to Mr. Rajesh and Mr. Uday (Department of Metallurgy and Material Science) for helping me for doing SEM and XRD analysis of my samples.

Finally, yet importantly, my sincere thanks to all my friends who have patiently extended all sorts of help for accomplishing this undertaking, and the successful completion of this project.

10th May, 2012

Thanking You, ANKITA BILUNG

CONTENTS

CERT	TIFICATE	
ACKI	NOWLEDGEMENT	
LIST	OF FIGURES AND TABLES	5
ABST	TRACT	6
I.	INTRODUCTION	7
	a. Physical Properties of Cordierite and Mullite	
	b. Cordierite-Mullite Composite	
	c. Properties	
	d. Application	
II.	LITERATURE REVIEW	11
III.	OBJECTIVE	18
IV.	EXPERIMENT WORK	19
	A. Synthesis of mullite- cordierite composite	
	B. Bulk Density and Porosity of sintered pellets	
	C. X-ray diffraction	
	D. Dilatometer	
	E. Biaxial flexural strength	
V.	RESULT AND DISCUSSION	
	A. XRD- charred rice husk	24
	- Pellets sintered at 1400 [°] C	
	B. Dilatometer reading	
	For 70 (%mullite): 30(% cordierite)	
	C. Theoretical density ~apparent porosity	
	D. Biaxial flexural strength	

VI.	CONCLUSION	32
VII.	FOR FURTHER STUDY	33
VII.	REFERENCES	34

LIST OF FIGURES

Figure 1: Flowchart of the experiment

Figure 2: Ternary phase diagram for alumina, silica, and magnesia system

Figure 3: XRD OF CHARRED RICE HUSK

Figure 4: XRD OF DIFFERENT COMPOSITIONS

Figure 5: DILATOMETER READING FOR 70:30 (mullite: cordierite) BATCH

Figure 6: THEORITICAL DENSITY~APPARENT POROSITY

6a: 70:30(mullite: cordierite)

6b: 30:70(mullite: cordierite)

6c: 50:50(mullite: cordierite)

Figure 7: BIAXIAL FLEXURAL STRENGTH

7a: 70:30 (mullite: cordierite wt %)

7b: 30:70 (mullite: cordierite wt %)

7c: 50:50 (mullite: cordierite wt %)

7d: %mullite in the composition vs. biaxial flexural strength

LIST OF TABLES

Table I: Physical Properties of Cordierite and Mullite

ABSTRACT

Cordierite-mullite composite has been prepared from rice husk derived silica; Magnesium sulphate derived magnesium oxide, and calcined alumina powder. Phase formation behavior of the sample has been studied from the XRD pattern of the sintered sample. The densification behaviour of the composite has been studied using dilatometer. The densification behavior of the composite as a function of sintering temperature has also been reported. Flexural strength as a function of sintering temperature and mullite content in the composite has also been studied.

I. INTRODUCTION

Cordierite (2MgO. 2Al₂O₃. 5SiO₂) ceramics have been well studied for its excellent property of thermal shock resistance, low thermal expansion coefficient and low thermal conductivity. The properties enable it to be used in places subjected to very rapid heating and cooling conditions. Low dielectric constant (ε =5–6), high resistivity (ρ >10¹² Ωcm), elevated thermal and chemical stabilities, very low thermal expansion coefficient (α =1–2×10⁻⁶ °C⁻¹) and low processing costs suggests it as a potentially available material that can replace alumina substrates, traditionally used in the electronic industry. It's typical applications includes refractory shapes, welding tapes, heater, thermocouples and appliance insulators etc.

Mullite $(3Al_2O_3, 2SiO_2)$ is used as structural materials because of its excellent mechanical properties at high temperatures. The raw materials for mullite are easily obtainable and are reasonably priced. It has excellent high temperature properties with improved thermal shock and thermal stress are attributed to the low thermal expansion, good strength and interlocking grain structure. The largest use of mullite based products is in refractories for glass and steel industries. The steel industry is the largest user, where refractoriness, high creep resistance and thermal shock resistance are important. The glass industry uses mullite based refractories in burner blocks, ports and in checker bricks as well as in the upper structure of the tanks where the glass is melted and in the drawing chambers. Thermal shock resistance, chemical attack resistance, high hot strength and creep resistance are the primary properties valued by the application industry. They are also used as protection tubes, furnace liners, electrical insulators etc.

The different physical properties has been tabulated in Table - 1

PROPERTIES	MULLITE	CORDIERITE
Chemical formula	3Al ₂ O ₃ . 2SiO ₂	2MgO.2Al ₂ O ₃ . 5SiO ₂
Density	3.03g/cc	2.60 g/cc
Young's modulus	130GPa	70GPa
Modulus Of Rupture	160MPa	117MPa
Thermal expansion coeff.	4.5- 5.6 (x10 ⁻⁶ / °C)	1.7(x10 ⁻⁶ /°C)
Thermal Conductivity	4-6 W/m K (100-1400°C)	3 W/m K (room temp.)
Max. Operating temp.	1725 °C	1371°C

|--|

Cordierite – Mullite Composites

Cordierite (2MgO. 2Al₂O₃. 5SiO₂) and mullite (3Al₂O₃. 2SiO₂) have become an important, worth noticing ceramic because of it wide range of applicability. It has been used as refractory materials because it can withstand the fast firing techniques for development of ceramic products. They have in recent years also attracted much attention as substrate material as cordierite has low average thermal expansion coefficient compared to Si chips where microcircuits carrying out the logic and memory functions. But the problem faced by cordierite materials is the difficulty to sinter by solid state process though any sintering aids can help in their densification by liquid-phase process. Unfortunately, it is due to these sintering aids that the electrical and thermal properties of cordierite are degraded.

Addition of mullite to cordierite allows tailoring of the thermal expansion coefficient of composites to match to that of Si, hence preventing chip detachment and device failure. Mullite morphology also is important for its applications, as there are two common morphologies for mullite. One is a platelet shape with low aspect ratio and the second is a needle shape with high aspect ratio. If the needle shape mullite can form in a ceramic body during sintering, it has an effect on both the mechanical and physical properties by increasing the mechanical strength and thermal shock resistance. The most important condition relates to ceramic chemical composition. If the silica and alumina ratio with low basic materials such as sodium and calcium is adjusted, the needle shape mullite forms at about 1400°C and the needles will interlock. This mechanical interlocking causes the porcelain to have high mechanical strength.

As Cordierite is a silico-aluminate of magnesium whose low thermal expansion gives it an excellent thermal shock resistance. Hence compositions made of Cordierite-Mullite can improve the creep at higher temperatures. These are generally used in Tubes, firing supports used in oxidizing or reducing conditions, especially recommended for the rapid firing.

Properties of Cordierite-Mullite Composites

The composite of cordierite-mullite, together as a material is also of high significance because each both individually support each other, hence bringing the best character out of it. Properties making it useable in various applications are:-

- Cordierite ceramics have the excellent property of thermal shock resistance. It enables them to be used under a severe environment of very rapid heating and cooling conditions.
- Mullite is used as structural materials due to its excellent mechanical properties even at high temperatures.
- The composite has a longer service life
- Excellent thermal shock resistance
- Light in volume

- High thermal stability
- Low thermal expansion and conductivity.
- Improved creep resistance at high temperature.
- Energy saving effect
- Cordierite has high bend strength due to the low thermal expansion, and has low sintering range, the presence of Mullite helps in widening the sintering range, increasing refractoriness of materials.
- Mullite has a higher operating temperature than cordierite but as a composite their Maximum operating temperature -1350 °C

Applications

Cordierite has been used in tubes, firing supports in oxidizing and reducing conditions and especially recommended for rapid firing purposes.

Mullite on the other hand is used in muffles and tubes, and as a support for firing and resistance.

But as composite cordierite- mullite are used as:-

- Especially an ideal material for heavy clay industries.
- Used in shuttle kilns, tunnel kilns and roller kilns.
- For high temperature filtration applications (i.e. as ceramic filters).
- As refractories in fast-firing techniques used for development of ceramic products.
- As electrical porcelains
- Catalytic converter substrates for exhaust gas control in automobiles
- Packing materials in electronic packing

II. LITERATURE REVIEW

In order to minimize stress during operation at silicon-substrate interface of an integrated circuit device *James.D.Hodge*, of *Cohoes*, *NY*^{{11}</sup> used mullite, as it had a thermal expansion coefficient which matched nearly that of silicon but as it also had to undergo heating and cooling cycles during the operation which is especially critical in semiconductor devices. Therefore the thermal expansion coefficient is made lower to match that of silicon through addition of second phase having low thermal expansion coefficient. Cordierite-mullite has an advantage over mulliteglass, as the former have higher thermal conductivity. At around 1290-1550°C sufficient liquid phase is generated to densify the body. Another reason this being used together is that both co-exist in thermal equilibrium i.e. mullite can exist in cordierite phase and cordierite can exist in thermal equilibrium with mullite phase.

Commercially available cordierite and mullite powders were used by *M.A. Camerucci* and *et.al* ^[2] to obtain cordierite-mullite composite materials with mullite content up to 65 wt.% . The cordierite powders were the coarse, medium and fine and contained in binary mixtures of 30, 50 and 70 weight percent of the smaller size component. The influence of the porosity, mullite and glassy phase contents and grain size on the electrical behavior of the composites has been analyzed. The thermal expansion behaviour of the composite as a function of the composition has also been reported.

Junichi Takahashi, *et.al.* ^[3] studied the effect of precursor materials on the morphology of mullite grains in cordierite-mullite composites. It has been reported

that the morphology of the mullite grains was substantially changed depending on the Al-source used during the fabrication of the composites. Acicular grains were reported to produce from the alumina sol-containing mixtures, whereas the growth of angular or granular mullite grains occurred for the mixtures containing $Al(OH)_3$ powder. Higher reactivity combined with better dispersability in precursor powders has been attributed to the lowering in the mullitization temperature in the composited prepared with alumina sol as compared to those containing $Al(OH)_3$ powder.

Cordierite-Mullite Composite has been prepared using commercially available cordierite and mullite powders as starting raw materials with various ratios up to 100 wt% by *Phatthamon Kiattisaksophon*, *Sukdiphon Thiansem* ^[4]. The precursors has been mixed together followed by ball milling to prepare the composite. After pressing it was sintered between 1250°C and 1400°C with a temperature interval of 50°C. The thermal expansion coefficient was measured and observed by loop of thermal behavior when heated up to maximum temperature and cooled down to room temperature. On studying the thermal behavior it was seen that the percentage of cordierite-mullite at 70:30 wt% sintered at 1400 °C showed good results in both of physical and thermal properties. It also showed better densification of the body and variation in the pore size. Bulk density is 2.58 g/cm³, apparent porosity is 1.00 % and water absorption is 0.39 %. Thermal expansion coefficient showed the value of 2.98 x 10⁻⁶ °C⁻¹ that is very low. For thermal shock resistance testing, it exhibited 8 cycles. Finally, The SEM photograph showed densification of the body containing cordierite and mullite structure. These affecting to improve thermal shock resistant and spot crack for the body. The orientation of mullite crystal in pores of body exhibited as the network. Cordierite prism was formed in mullite matrix and melted in glass phase, filled in

pores of mullite crystal structure. The pores of body improved thermal behavior. When the materials received thermal stress, it could reduce thermal stress by pores and keep the deformation of materials.

Cordierite-mullite compositions used as substrates in fast firing of porcelain whiteware are characterized by different microstructure morphologies and crack propagation behaviour. The characterization of thermal-shock degradation of commercial refractories in service is of prime concern to the industry involving high temperature processes. When refractories are subjected to industrial thermal cycles crack nucleation and propagation occurs resulting in loss of stiffness, mechanical strength and degradation. The development and use of higher quality raw materials has increased the cost of refractories and led to consider the use of non-destructive testing methods for characterizing thermal shock damage. D. N. Boccaccini et.al^[5] studied the thermal degradation of commercially available cordierite-mullite refractory materials when subjected to industrial thermal cycles by comparing the degradation curves obtained from non-destructive testing methods, e.g. ultrasonic velocity testing (UPVT) and image analysis, with those obtained from flexural strength and fracture toughness measurement by three point bending test and chevron notched technique, respectively. The measurement of the ultrasonic velocity was used to assess the material degradation with increasing thermal shock cycles and specimen damage was monitored using image analysis and further results of material degradation were obtained. The correlation between thermo-mechanical properties, microstructure, crack propagation behaviour and thermal shock resistance was checked to make, service life prediction models of refractory plates from measured values of ultrasonic velocities in plates.

Mullite has achieved outstanding importance as a material for both traditional and advanced ceramics because of its favourable thermal and mechanical properties. Due to its high temperature but low pressure formation conditions, mullite occurs very rarely in nature. The outstanding scientific and technical importance of mullite as be explained by *H. Schneider et.al*^[6],

- Its high thermal stability and the favorable properties like low thermal expansion and conductivity, high creep resistance and corrosion stability together with suitable strength and fracture toughness.
- The fact that the starting materials (e.g., alpha-alumina plus silica, alumino silicates of the composition Al₂SiO₅, i.e. sillimanite, and alusite and kyanite, refractory-grade bauxite, Al₂O₃-rich sheet silicates and clays) are available in big quantities on earth. Thereby kaolinite and other clay-based materials achieved high importance, since they allow multiple shaping procedures of components and structures in the green state.
- Its ability to form mixed crystals in a wide Al2O3/SiO2 range and to incorporate a large variety of foreign cations into the structure.
- The fact that the structural principles of mullite senso stricto can be extended to a large number of phases belonging to the family of mullite-type structures.

Three types of polycrystalline mullite ceramics may be distinguished: *monolithic mullite ceramics, mullite coatings and mullite matrix composites.*

Sinter-mullites are produced by heat treatment of the starting materials, essentially via solid-state reactions. These mullites tend to have "stoichiometric", i.e., 3/2-composition ($3Al_2O_3 \cdot 2SiO_2$, i.e., ≈ 72 wt. % Al_2O_3).

Fused-mullites, produced by crystallizing melt of alumino-silicate. These mullites tend to be Al_2O_3 -rich with approx. 2/1-composition ($2Al_2O_3 \cdot SiO_2$, i.e., ≈ 78 wt. % Al_2O_3)

Chemical-mullite are produced by heat treatment of organic or inorganic precursors. The composition of such mullites strongly depends on the starting materials and the temperature treatments. Extremely Al_2O_3 -rich compounds (>90 wt. % Al_2O_3) have been identified at synthesis temperatures <1000 °C.

Variety of routes have been used for their manufacture such as using mixed, premade mullite and cordierite powders, mullite powder and cordierite composition glass, combustion synthesis and mixed mullite and cordierite mixing powders and routes based on sol-gel precursors by considering the complex interrelations between processing, microstructure and electrical and mechanical properties for mullite-cordierite composites. Applications of this composite are not just restricted to high-temperature properties but its excellent electrical insulating ability makes them potentially useful in applications ranging from refractories to computer substrates. In their study, *T. Ebadzadeh and W. E. Lee*^[7] prepared mullitecordierite composites by two methods:

(1) From a mixture of precursors, and

(2) From a mixture of pre-made mullite and cordierite powders.

Calcining the precursor powders at low temperatures (600 and 950°C) to facilitate densification due to the presence of highly reactive phases such as *gamma* alumina and amorphous silica and sintering via viscous flow. Crystallisation, densification behaviour, and microstructural development of mullite-cordierite composites prepared from these different starting materials were examined. Mechanical and dielectric properties were measured and related to the observed microstructures. Crystallisation occurred more readily in mullite-cordierite composites made from

powders derived than from aluminum sulphate precursors due to the finer scale of mixing of the starting materials but crystallisation also leads to lower densities because densification by viscous flow is more limited. Composites made from precursor derived composites achieve higher density at lower temperatures due to viscous flow of the glass present in them. And hence relating the mechanical properties to the microstructures and their development through processing. It is seen that strength and hardness data could only be sensibly understood by correlating to composition, grain size and porosity measurements. Toughness values were all about 1.5-2MPam^{1/2}. Dielectric data scaled well with the mixing rules for composites if normalised for porosity content.

Ability to control macroscopic properties such as thermal expansion and fracture strength of ceramic materials have significant benefits for numerous technologies, especially when materials are, required to withstand high stresses as well as with severe thermal gradients. In dense materials, desirable values of thermal expansion and other properties such as strength and modulus are accomplished by making physical mixtures of matrices with desired amounts of embedded fibers or particles of carefully chosen characteristic properties. Fibers or particles reinforce the matrix and affect its modulus or thermal expansion characteristic. The preparation of porous ceramic materials with targeted thermal expansion characteristics as done by *Daniel Grohol, et.al.*^[8] was achieved by similar means as done in dense materials, except that pores were created by the addition and subsequent burnout of sacrificial porogen. It can also be done by coating of polymer foams of desired pore structures in preceramic slurries has been used followed by the removal of templating foam.

Solid-state chemical reaction was used, during which one ceramic material with its set of desirable properties is partially converted into another material with another

set of desirable properties. The resulting composite then adopts the advantages of both its parent components. In the prepared series of cordierite-mullite composites, coefficient of thermal expansion values decreased as the cordierite content increased. The composites were synthesized in two steps: in the first step, interconnecting acicular mullite grains with a truss-like structure were formed in the presence of SiF₄ at elevated temperature; the mullite framework was interspersed with particles of MgF₂ and SiO₂. In the second step, varying amounts of the mullite framework were converted into cordierite; MgF₂ and SiO₂ particles reacted with parts of the mullite framework that also served as a structural template. Near-linear dependence of coefficient of thermal expansion on cordierite content indicated that discontinuous high- coefficient of thermal expansion mullite phase was interrupted by the low- coefficient of thermal expansion cordierite phase essentially in the entire mullite-cordierite composition range. This makes the acicular mullite-cordierite composites gives easy control over technologically important material attributes such as fracture strength and resistance to thermal shock.

Cordierite–mullite percentage was kept constant (70:30%).cordierite supplies resistance to shock, while mullite provides the strength needed. The presence of cordierite lowers the reaction temperature and decreases the firing shrinkage. Both mullite and cordierite were added in different proportions either as grog or raw material by *D.M.Ibrahim et.al*.^[9] and the effect of the added grog percentage on body properties was seen. The best mechanical, thermal and physical properties are achieved with bodies having 50-70% grog. Super-stoichiometry of alumina, added in the form of mullite grog or calcined bauxite improved both physical and mechanical properties, without affecting spalling resistance. Mullite addition also raised the mechanical properties and refractoriness underload of the mixes.

III.OBJECTIVE

- Synthesis of pure silica from rice husk.
- Synthesis of pure cordierite-mullite composite using the synthesized silica from rice husk, magnesium sulphate and calcined alumina as precursor materials.
- Study the densification behavior and strength of the composite.

IV. EXPERIMENTAL WORK

A. Synthesis of mullite- cordierite composite

The raw materials used are:

- Rice husk for silica
- Magnesium sulphate for magnesia
- Calcined alumina

Preparation of Silica from rice husk:

- i. The rice husk was washed thoroughly with water 5-6 times in a trough.
- ii. Then it was allowed to dry.
- iii. Then the dried husk is taken in batches of 50gm, and for each batch 375 ml of water and 125ml of hydrochloric acid is taken and proceed for leaching process.
- iv. Leaching- this is done to remove Na, K, Ca ions present in the husk.
- v. The H_2O and HCl is mixed in a beaker and heated.
- vi. Just when it starts boiling 50gm of the husk is added into it. Boiling is continued, till a chocolate brown colour is seen. (note the level of H_2O and HCl should be maintained throughout the heating process)
- vii. Now add boiling water to the leached rice husk, and then rinse the water out, continue the process until a pH 7 is obtained.
- viii. Dry it, and then put it for calcination at 700°C for a soaking period of 4 hours.
 - ix. The rice husk chars off, leaving behind SiO₂.
 - x. This SiO₂ is grinded with the help of mortar and pestle, and kept for further use.

FLOWCHART OF THE EXPERIMENTS



Fig 1: flowchart of the experiment

a. Batch preparation

Mullite and cordierite is taken in 70:30; 50:50; and 30:70 weight percent ratios respectively. For mullite: SiO_2 , Al_2O_3

For cordierite: SiO₂, Al₂O₃, MgO

The raw materials required for both mullite and cordierite, magnesium sulphate is added in the form of solution, and they are mixed together. Allow each of the batch to dry.

b. Calcination – the dried sample is then calcined at 900°C, for a soaking period of 2 hours.

c. Pellets preparation – as the pellets had to be sintered at 5 different temperatures, therefore 5 pellets of 0.5g were made for each temperature, hence 25 pellets for each composition.

To the 0.5g of sample, 3% PVA was added and then pressed at 3tonnes of pressure in the *Carvur Press*.

d. Sintering – then the pellets were sintered at different temperatures, $1200 \,{}^{0}$ C, $1250 \,{}^{0}$ C, $1300 \,{}^{0}$ C, $1350 \,{}^{0}$ C, $1400 \,{}^{0}$ C.



Fig 2: ternary phase diagram for alumina, silica, and magnesia system.

B. Bulk Density and Porosity of sintered pellets

The bulk density and apparent porosity of the sintered pellets were determined by Archimedes principle using water. Dry Weight is measured and then the pellets were put in water and boiled for about 30 min-45 mins. After that suspended weight is measured using apparatus in which pellet is suspended in water and weight is measured. After taking suspended weight, soaked weight is taken. Hence the dry weight, soaked weight and suspended weight were measured.

The bulk density and apparent porosity were calculated by the formulas:

Bulk Density = dry weight / (soaked weight – suspended weight) Apparent porosity= (soaked weight-dry weight) / (soaked weight – suspended weight)

C. X-ray diffraction

The X-ray diffraction of the pellets sintered at 1400 ^oC was performed in **PW1830 diffractometer**, (**Phillips**, **Netherland**) at a **0.04 scan rate** from **20-80^o** for **25 minutes**. This is done to know the different phases present in the pellet.

D. Dilatometer

0.7 g of sample was taken and pressed in the form of a bar. It was put in the dilatometer, to study the shrinkage behaviour upto a temperature of 1300° C

E. Biaxial flexural strength

In this the pellet is kept in such a way that it can roll along its thickness. The pellet is made to stand and pressure is applied to it, and the force at which it breaks or cracks is noted down. However prior to it, the thickness and the diameter are measured and noted down.

To find the compressive strength we use the formula:

 $CCS = (2 \times Force)$

(Pi) x diameter x thickness

V. RESULTS AND DISCUSSION

A. X-Ray Diffraction



Fig3: XRD pattern of charred rice husk

The XRD pattern of the charged rice husk has been shown in Figure 3. It could be seen from the pattern that the powder is mostly amorphous in nature.

However, the broad peak around 21.8 may be attributed to crystalline silica.



Fig 4: XRD pattern for composite of different compositions

The XRD pattern of different cordierite-mullite composite sample sintered at 1400° C, for 4 hours has been shown in Figure 4 where in A- Represents the sample with composition 30% mullite and 70% cordierite, B- Represents the sample with composition 70% mullite and 30% cordierite and C- Represents the sample with composition 50% mullite and 50% cordierite.

All the samples show the presence of cordierite and mullite phases only. No other impurity could be detected in the X-ray Diffractogram. A closer observation on the XRD pattern shows that the highest mullite peak intensity changes gradually with increase in mullite content in the sample. So this is in accordance with the reaction proposed for the synthesis of the composition.

B. DILATOMETER



Figure 5: Dilatometer reading for 70:30 (mullite: cordierite) batch

The typical densification behavior of 70% mullite and 30% cordierite (weight %) composite is shown in Figure 5. It could be seen that upto 800° C, no substantial shrinkage occurs in the

composite. The onset of densification was found to be 810° C. Rapid shrinkage observed in the temperature range beyond 800° C is attributed to the liquid phase densification in the sample.

The densification has not been completed in the studied temperature zone. All the other composites require also showed similar behavior.

C. %THEORETICAL DENSITY ~ APPARENT POROSITY



Fig6a: 30:70 (%cordierite: % mullite)



Fig 6b: 70:30 (%cordierite: % mullite)



Fig 6c: 50:50(%cordierite: % mullite)

Apparent porosity and bulk density as a function of sintering temperature for the composites has been shown in Figure 6 (a-c) wherein *a indicates 70% mullite and 30% cordierite (weight %) composition, b- Indicates 30% mullite and 70% cordierite (weight %) composition and c-Indicates 50% mullite and 50% cordierite (weight %) composition*

All the composites show that the density of the sample increases with sintering temperature. The increase in density with increase in temperature is due to increase in liquid content in the sample at higher temperature which results in enhanced densification in the sample.

The apparent porosity of the sample decreases with increase in the temperature, which is quite obvious. It could also be seen that 50:50(%cordierite: % mullite) showed enhanced densification as compared to the other two.



D. CCS (Bi axial flexural strength)

Figure 7a: 70:30 (mullite: cordierite wt %)



Figure 7b: 30:70 (mullite: cordierite wt %)



Figure 7c: 50:50 (mullite: cordierite wt %)



Figure7 d: % mullite in the composition ~ biaxial flexural strength

Bi-axial flexural strength of the sample is shown in Figure 7. Wherein *a- indicates 70:30* (*mullite: cordierite wt %*), *b- Indicates 30:70* (*mullite: cordierite wt %*), *c- Indicates 50:50* (*mullite: cordierite wt %*) and *d- Indicates %mullite in the composition ~ biaxial flexural* strength

All the composition shows an increase in the strength, with increase in temperature could be correlated with the porosity of the sample. As the sintering temperature increases, the porosity decreases. Decrease in the porosity is related to increase the strength of the sample.

Figure 7d shows the variation of bi-axial flexural strength as a function of mullite content in the composite. It could be seen that the bi-axial flexural strength of the sample could be correlated with the interlocking matrix by mullite grains. Increase in the mullite content in the composite

provides better interlocking microstructure and thus enhances the bi-axial flexural strength in the sample.

CONCLUSION

The following conclusions can be drawn from the present study:

- All the samples give phase pure cordierite and mullite.
- The density of the sample increases with increase in sintering temperature and is governed by liquid phase sintering.
- The flexural strength increases with increase in porosity of the sample.
- Increase in mullite content of the composite, results better strength in the composite.

For further study....

- Detail phase formation behavior need to be studied.
- Microstructure evaluation needs to be studied to correlate the strength.
- Thermal expansion behaviour needs to be studied.

REFERENCES

1. James D.Hodge, Cohes, N.Y "**Mullite-cordierite composite ceramic and method for preparation**", United states Patent; patent number: 4,528,275

2. M.A. Camerucci ,G. Urretavizcaya , M.S. Castro, and A.L. Cavalieri, "Electrical properties and thermal expansion of cordierite and cordierite-mullite materials", Journal of the European Ceramic Society 21 (2001) 2917–2923

3. Junichi Takahashi, Maya Natsuisaka, and Shiro Shimada **"Fabrication of cordierite–mullite** ceramic composites with differently shaped mullite grains", Journal of the European Ceramic Society 22 (2002) 479–485

4. Phatthamon Kiattisaksophon and Sukdiphon Thiansem, "The Preparation of Cordierite-Mullite Composite for Thermal Shock Resistance Material", Chiang Mai J. Sci. 2008; 35(1):
6-10

5. N. Boccaccini, Elie Kamseu, T. D. Volkov-Husović, M. Cannio, M. Romagnoli, P.
Veronesi, C. Leonelli, I. Dlouhy, and A. R. Boccaccini., "Characterization of thermal shock damage in cordierite-mullite refractory materials by non-destructive methods" Association of Metallurgical Engineers of Serbia (AMES), Review paper UDC:669.162.212.8:661.962.1=20

6. H. Schneider , J. Schreuer, and B. Hildmann, "Structure and properties of mullite—A review", Journal of the European Ceramic Society 28 (2008) 329–344

7. T. Ebadzadeh and W. E. Lee, "**Processing-Microstructure-Property Relations in Mullite-Cordierite Composites**", Journal of European Ceramic society 18(1998) 837-848

8. Grohol, D., Han, C., Pyzik, A. J., Goss, J. M. and Todd, C. S. (2010), "Acicular Mullite– Cordierite Composites with Controllable CTE Values", Journal of the American Ceramic Society, 93: 3600–3603 9. D.M Ibrahim, S.M.Naga, Z.Abdel Kader and E.Abdel Salam, "Cordierite-mullite refractories, "Ceramics International 21(1995) 265-269

10. Precipitated Silica from Rice Husk Ash IPSIT (Indian Institute of Science Precipitated Silica Technology)