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Effect of Sintering At Different Temperature to Enhance the Physical and Mechanical Properties of Forsterite Refractories Doped With Kaolin

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ABSTRACT: Forsterite (Mg₂SiO₄) one of the most important refractory materials which are being extensively used in steel plants and iron industries. The present study investigates effect of sintering at different temperature to enhance the physical and mechanical properties of forsterite refractories doped with kaolin were done. It was found that the bulk density, bending strength and compressive strength increased with increasing kaolin content, up to 2 to 4%. Further, as the kaolin content is increased to 6% it starts to decrease due to formation of more mullite phase. Percentage apparent porosity decreases with increases the kaolin concentration. Each composition sintering temperatures were done at 1200, 1300 and 1400°C having soaking time for 3 hour. XRD and SEM analysis was done to the sintered forsterite. The forsterite crystals were developed after heat treatment were deeply analyzed as per there uses as a refractory materials and it is found that forsterite with kaolin content of 2-4% serves as better refractory material than pure forsterite.

KEYWORDS: Forsterite, kaolin, mullite, bulk density, bending strength compressive strength

I. INTRODUCTION

Refractory material is that material which withstand at very high temperature without losing it physical and chemical property. They have good creep resistance and high refractoriness temperature under load [1]. Refractory materials are used as linings for furnaces, incinerators, reactors and also used to make crucible and moulds for casting glass and for surfacing flame deflector systems for rocket launch structures [2]. Today, in iron-and steel-industry utilizes roughly 70% refractory material [3]. Having such properties, the results of forsterite can be utilized as a part of smelting, glassmaking, and rotary cement kiln [4, 5]. In refractory fields, forsterite is considered as one of the most efficient materials for critical production stages of modern iron and steel industry, namely, steel making and casting or ladle metallurgy [6-11]. It is a kind of thermal insulation and heat preservation refractory because its heat conductivity is low (about 1/3-1/4 of the pure MgO) (Xu and Wei, 2005). Use of forsterite ceramic material at high frequency application, having high coefficient of thermal expansion, packing materials in electronic packing, refractory coating on metals, integrated circuit substrates, etc. [12-13]. The evolution and growth of Forsterite ceramics was manifested over the last few decades to improve the physic mechanical properties; strength, dielectric constant, thermal expansion, thermal conductivity and mechanical performance. The main objective of the work is to increase its bulk density and compressive strength by addition of kaolin. Kaolinite is a clay mineral having chemical composition Al₂Si₂O₅(OH)₄. The Rocks that are rich in kaolinite are known as kaolin or china clay [14]. The present study deals with the synthesis and characterization of forsterite using kaolin as a doping like 2%, 4% and 6%, and check the physical property ie. apparent porosity, bulk density, linear shrinkage and mechanical properties like compressive strength and bending strength of the forsterite at different temperature like 1200, 1300 and 1400°C with soaking time of 3 hour.



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II. **EXPERIMENTAL**

Material Preparation: Prepare 100g of mixture in which 56 MgO (from Thomas baker chemicals pvt. Limited Mumbai) and 44 SiO₂ (From Loba Chemie Laboratory and fine chemicals) wt% were mixed with different concentration of kaolin. Concentrations of kaolin were taken as 2, 4 and 6 wt% were doped with replacing concentration of SiO₂. Proper mixing was done for 5 to 6 hours. The moisture removed from the mixture, after dried at $105 + -5^{\circ}$ C for one day. After drying the mixture 2% of PVA were added as a binder again mix it properly to make it homogeneous. The mixture was compacted by hydraulic pressing machine applying pressure of 15 ton in 15 mm die. The pellets were prepared by pressed for a constant time period of 5 minute (Holding time). The compacted powder (pellets) thus formed are then sintered at different temperatures, temperatures being 1200, 1300 and 1400^oC. Duration of heating at peak temperature for each sample the soaking time is 3 hour.

III. RESULTS AND DISCUSSION

In order to check the characterization behaviour and properties of forsterite as a refractory material on addition of different percentage of kaolin. Different physical and mechanical properties like bulk density, apparent porosity, linear shrinkage and mechanical properties like compressive strength and bending strength after sintering were calculated for every sample and the data thus obtained are deeply analysed for the trend of this properties as to how they vary on addition on kaolin with sintering temperature having soaking time for 3 hour.

1. Compaction and Sintering: The ball milled and dried powder is compacted into green samples of similar diameter and thickness. These obtained green samples were sintered at 1200, 1300 and 1400^oC for 3 hour. In this temperature range sintering process has taken place which is justified by shrinkage in volume of samples.

2. **Bulk Density Measurement (B.D):** Bulk density is defined as the dry weight of sample per unit volume of sample. Bulk density considers both the solids and the pore space. The densities of the samples sintered at different temperatures for different soaking time are calculated.

Formula for bulk density can be written as B.D= Dry weight/(wet weight- Suspended weight)

The bulk densities obtained for all samples were found to vary from 2.05 gm/cc for sample having 0% kaolin content which is sintered at 1200° C to 2.64 gm/cc for sample having 4% kaolin content sintered at 1400°C. Density is found to increase with increase in sintering temperature. And it is maximum for sample which sintered at 1400°C soaking time is 3 hour . After addition of 6% kaolin its bulk density slightly decreases up to 2.58 gm/cc sintered at 1400° C (shown in figure 1).



Figure1: Graph between Bulk Density and kaolin (0 to 6%) as doped sintered at different temperature with soaking time for 3 hour.

3. Apparent Porosity Measurement (A.P): Porosity or void fraction is a measure of the void (i.e. "empty") spaces in a material, and is a fraction of the volume of voids over the total volume. The formula for porosity can be written as A.P = (W-D/W-S)*100.



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Where W is the weight, D is the dry weight, S is the suspended weight. From the figure 2 it is found that as kaolin concentration is increased apparent porosity decrease till 4% (concentration of kaolin) after that it starts to increase.



Figure 2. Graph between Apparent Porosity (in %) and kaolin (0 to 6%) as doped sintered at different temperature with soaking time for 3 hour.

4. Linear Shrinkage: Linear shrinkage for a sample with concentration of kaolin increases with increases in sintering temperature having soaking time for 3 hour. From the figure 3 it is found that as the kaolin concentration is increased linear shrinkage also increases till 4% (concentration of kaolin) after more addition of kaolin it starts to reduce.



Figure 3. Graph between Linear Shrinkage (in %) and kaolin (0 to 6%) as doped sintered at different temperature with soaking time for 3 hour.

5. Compressive Strength: In the study of strength of materials, the compressive strength is the capacity of a material to withstand loads tending to reduce size. It can be measured by plotting applied force against deformation in a testing machine. The Study was made of the relationship between the compressive strength (σ comp) of fotsterite and temperature. The specimens were made of quartz and magnesium oxide powder and 2 to 6% of kaolin as an additive for increasing the compressive strength. The pure specimens were pressed at 510 kg/cm² which is fired in a silicon carbide furnace at 1100°C with a soak of 3 h. At 1400°C compressive strength (σ) is 617 (kg/cm²), show the characteristics of the fired specimens are given in figure 4. By further addition of 6% of kaolin in forsterite the compressive strength (σ comp) decreases from 617 to 602 kg/cm² because of porosity increases (Shown in figure 4).



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Figure 4. Compressive strength (σ =kg/cm²) of forsterite doped with kaolin content from 2 to 6% at different temperatures with soaking time for 3 hour.

6. Bending Strength: For bending test determination we prepare different samples having concentration of kaolin (0 to 6%) doped and sintered it at different temperatures. A rectangular specimen is required for the three-point bending test. The specimen is 4 centimeters long, 10 millimeters wide (b) and 10 millimeters thickness (d). The specimen is placed on two supports that are 2 cm apart (L), and the force (F) applying in the exact middle of the two supports (L/2). Shown in figure 5, it reveal that the bending strength of pure forsterite at 1200°C is 56 MPa after addition of kaolin concentration up to 4% it increases to 102 MPa which is sintered at 1400°C. For further doped ie. 6% it decreases due to porosity increases.



Figure 5. Bending strength of forsterite doped with kaolin content from 2 to 6% at different temperatures with soaking time for 3 hour.

7. X-Ray Diffraction (XRD) Analysis: Figure 6 shows X-ray Diffraction pattern of forsterite mixed with kaolin. The XRD patterns were recorded in the range of $10-80^{\circ}$ (2 θ). In Figure 6 displayed the formation of mullite on firing at different temperature with soaking time of 3 hr. All the peaks characterizing forsterite (JCPDF #34-0189 & #34-0189) and mullite (JCPDF # 15-0776) are detected. No peaks characterizing of any other phase are detected.



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Figure 6. X- ray diffraction patterns between 20 to 80° samples fired at 1200, 1300 and 1400°C.phases are identified by the following symbols: M= Mullite, F= Fosterite

8. Scanning Electron Microscopy (SEM) Analysis: After firing a sample at 1400°C with soaking of 3 hour, from the SEM analysis it reveal that there is a formation of large mullite crystals with higher aspect ratio needle corresponding to primary mullite develop in a glassy matrix shown in figure 7 and 8. The mullite can be formed at a temperature as low as 1100°C. The mullite needles show preferred orientation which help to lead the mechanical and thermal shock resistance. At higher temperature with soaking time smaller secondary mullite crystals nucleated by disintegration of alumina (in figure 8).



Figure 7. SEM analysis of forsterite doped with kaolin sintered at 1300°C with soaking time for 3 hour.



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Figure 8. SEM analysis of forsterite doped with kaolin sintered at 1400°C with soaking time for 3 hour.

IV. CONCLUSION

The forsterite crystals were developed after heat treatment were deeply analysed as per there uses as a refractory materials and it is found that forsterite with kaolin content of 2-4% serves as better refractory material than pure forsterite. The physical properties and mechanical properties of the forsterite have been done. The test was done for sintering temperature at 1200, 1300 and 1400°C with fixed soaking time for 3 hour.

- 1. The bulk density, compressive strength and bending strength of the forsterite material were increases with increasing concentration of kaolin up to 4%. Further, as the kaolin content is increased to 6% it starts to decrease due to formation of more mullite phase and increasing porosity. It is low without doped of kaolin into the composition.
- 2. Apparent porosity decreases and percentage linear shrinkage increases with increasing sintering temperature with the soaking period of 3 hour.
- 3. From the XRD analysis conforms that different crystal was develop after heat treatment with various sintering temperature.

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