



# Effect of MgO on the Sintering Behavior of Biphasic Alumina

## KEYWORDS

Reactive Alumina; Magnesia; Sintering, XRD.

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**ABSTRACT** Role of activated alumina, prepared by thermal dehydration of aluminium hydroxide on the sintering behavior of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> has been studied in relation to MgO addition as dopant. At the initial stage grain boundary movement was accelerated due to presence of activated alumina. The role of MgO did not deviate as that with single phase  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>.

## 1. Introduction

Alumina is the most widely used ceramic material for variety of engineering applications [1-3]. The most important properties of sintered alumina are high temperature stability, high hardness, strength, wear resistance, high thermal shock resistance. Sinterability of alumina powder is strongly influenced by the chemical and morphological homogeneity. Moreover particle size distribution, reactivity and the agglomerate nature play a vital role.

Radonjic, Nicolic and Obrenovic[4] studied the effect of original boehmite seeding and MgO on densification of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> and other phase transformations of transitional alumina. The boehmite structure plays a secondary role in all stages.

Bodur, C. T[5] investigated the pressureless sintering of MgO doped alumina for 3 to 6 hours and found that full densification did not occur at 1600°C.

Sone et al[6] observed that during sintering at 1600°C grain growth at surface region was inhibited upto one hour sintering but subsequently abnormal grain growth occurred.

Katsuda and Takashi[7] studied the sintering behavior of two  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> prepared respectively by Bayer process and alkoxide route and found differences in the plot of firing temperature and fired density.

Lotova and Dobrolyubov[8] investigated the sintering of alumina both pure and modified by compound additives at 1200 and 1500°C, using a structural energy parameter to assess the process and they offered a closer view of the sintering mechanism.

Sintering of alumina with respect to different parameter has been studied by many workers[9-12]. Activated alumina is of its amorphous mesophase transition[13]. The formation of activated alumina by thermal treatment is the result of transition phase of alumina with high surface area[14].

With the objective of enhancing the densification of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> compact this reactivity of transitional alumina phase has been utilized.

## 2. Experimental

$\gamma$ -Al<sub>2</sub>O<sub>3</sub> of very fine particle size was collected from Associ-

ated Cement Co. Ltd. in very pure form. Analytical grade MgO was selected to be used as a dopant.

Activated alumina was prepared by heating aluminium hydroxide gel at a temperature of 500°C for a period of 45 minutes at a heating rate of 5°C per minute.

Requisite proportion of the ingredients were taken in a ball mill with addition of 40% water and subjected to wet milling for a period of 16 hours. Then the mixed suspension was dried at 100°C which was again milled to dislodge the secondary agglomeration.

Rectangular bars of size 50mm x 5mm x 5mm were fabricated by uniaxial pressing at 1200 kg/cm<sup>2</sup>. Sintering was performed in slightly oxidizing atmosphere at temperatures from 1400 to 1650°C for a fixed soaking period of 2 hours.

The crystalline phases in the sintering specimens were identified by X-ray diffraction analysis using a Philips PW (1790) x-ray diffractometer.

The evolved microstructure was analysed through scanning electron microscope Model S-530, Hitachi.

## 3. Discussion

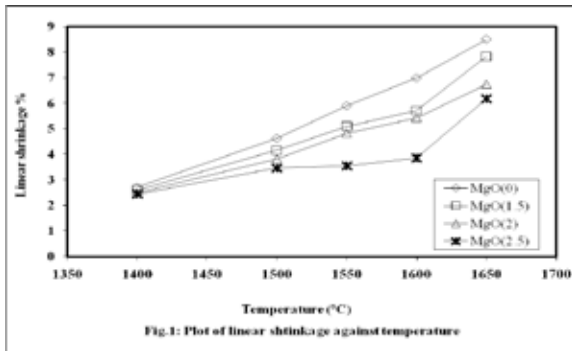
The purity of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> sample was ascertained through standard chemical analysis. The Na<sub>2</sub>O, Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> content which are harmful impurities were less. Al<sub>2</sub>O<sub>3</sub> content was 98.92%. Average particle size of the experimental  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> powder was 5.72  $\mu$ m.

Another ingredient was activated alumina whose surface area was found to be 150m<sup>2</sup>/g and the average particle size was 5.05  $\mu$ m. Chemical analysis indicated that it contained 92% alumina.

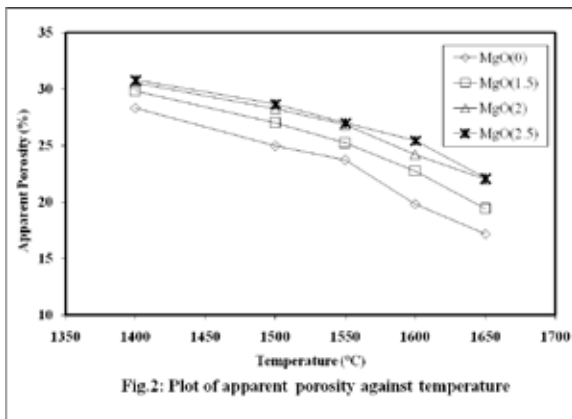
15% reactive alumina was added to  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> powder in the batch mixture followed by addition of different amounts of MgO. Activated alumina forms a stable green matrix improving the sinterability of the system. At elevated temperature it forms a bond at the interface.

The pressed bars were subjected to sintering in normal oxidizing atmosphere. The shrinkage during heat treatment was associated with the removal of residual water from alumina lattice and also densification of the compacted mass. The shrinkage curve followed a discontinuous path (Fig.1) having sharp inflection point and specially from 1600°C the

rate was significantly high. Maximum shrinkage under this condition was about 8.5%. Moreover the addition of dopant significantly influenced the shrinkage behavior as revealed from the experimental results. It is to be noted here that in presence of activated alumina the material transport took place at a rather faster rate because of less particle agglomeration. The initial nature of the curve with 2.5% MgO addition was different from the others.



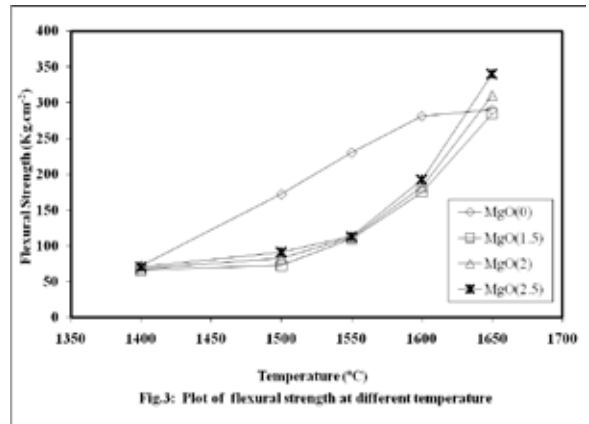
In this particular system densification proceeds through alignment of the developed grains and such pore evolution was in between the grains. In general porosity decreased with temperature in a step wise manner showing an inflection at 1550°C irrespective of the batch composition (Fig. 2) which appears to be related to the original batch mixture of both  $\gamma$ - $Al_2O_3$  and activated alumina. In general MgO addition to a certain point reduced grain growth and allowed sintering to proceed to a positive direction and that was also maintained in biphasic alumina system. MgO is known to eliminate discontinuous grain growth, suppress pore-grain boundary separation and decreases the grain growth.



In this case grain boundary migration is suppressed by the solid solution formation of MgO in alumina which has been enhanced by the presence of activated alumina.

For structural application of sintered  $Al_2O_3$ , change of mechanical properties is of great importance. Flexural strength of the sintered bars followed a direct relationship with temperature with or without addition of MgO (Fig. 3). The trend of the increasing path with MgO addition was different from that without MgO. In the latter case above 1600°C there was no increase of flexural strength. In other cases a sharp inflection was observed at 1600°C. Alumina is basically anisotropic in nature. Grain size and porosity have significant effect on flexural strength. Here also the phase transformation of activated alumina appears to be a

contributory factor for enhanced interlocking of the formed grains.



The major crystalline phase of the samples sintered at 1650°C was found to be  $\gamma$ - $Al_2O_3$  and the minor phase was only  $MgAl_2O_4$  with no other phase of  $Al_2O_3$  (Fig. 4).

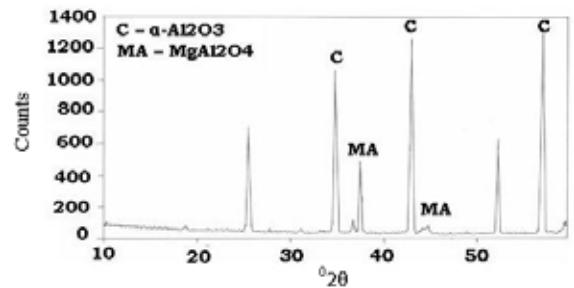


Fig. 4: X-ray Diffraction pattern of batch -III sample fired at 1650°C

The evolution of microstructure of biphasic alumina in absence and presence of MgO at 1650°C has been presented in Fig. 5. MgO addition helped in the development of crop of grains with well developed duplex structure.

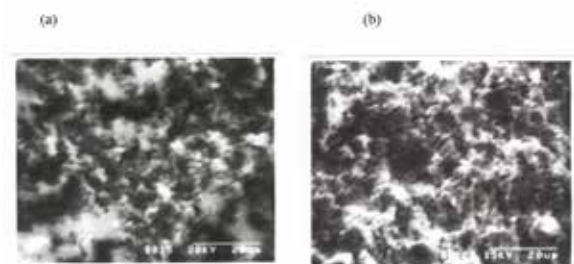


Fig. 5: Scanning Electron Microscopy of the sample sintered at 1650°C. (a) batch III and (b) batch IV

Batch IV with 2.5 % MgO showed extensive neck growth and inter connected grains with porosity remaining all along the edges, thus making a three dimensional network of pores. The remarkable feature of the microstructure was the rapid growth of few grains indicating high grain boundary mobility as initiated by activated alumina. Formation of  $MgAl_2O_4$  spinel phase favors inhibition of grain growth where as at the initial stage of sintering particle migration was accelerated by activated alumina resulting the retention of residual porosity. Another feature in this system was uneven distribution of grains which might be re-

lated to the deference in reaction rate of biphasic alumina with MgO. Activated alumina acted as a sintering aid but it was accompanied by slight grain growth. Discontinuous grain growth on the other hand was eliminated by MgO.

#### 4. Conclusion:

1. Activated alumina forms a stable green matrix improving the sinterability of the system. At elevated temperature it forms a bond at the interface.
2. Sintering of  $\gamma$ - $\text{Al}_2\text{O}_3$  in presence of activated  $\text{Al}_2\text{O}_3$  improved densification.
3. grain boundary migration is suppressed by the solid solution formation of MgO in alumina which has been enhanced by the presence of activated alumina.

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