Study on Sintering Characteristics of Alumina

P. VENU GOPAL[1], SHAIK BILAL[2], KHAJA ZAINUDDIN ANSARI[3], MOHD SHOEB AHMED[4], J. S. SONI[5]

[1]Assistant Professor, [2-4]Students, B.Tech, [5]Professor
Department of Mechanical Engineering
Bharat Institute of Engineering and Technology
Ibrahimpatnam- 501 510, Hyderabad, Telangana
INDIA

Abstract— Alumina is a ceramic material having high melting temperatures, good mechanical properties and it is used for various applications such as furnace components, electronics substrates, cutting tools, abrasion and corrosion resistant applications. The major intent of this work is to determine the sintering conditions required for densification of alumina powder to its near theoretical density. The effect of sintering temperature on the sintered density was studied in this paper. The effect of sintering time on densification was also studied by varying the sintering (soaking) time and found that the densification increases with increase in sintering time. Even though density increased with soaking time full densification was not achieved even at 14 hrs of sintering. For keeping sintering time within a reasonable limit, alumina samples were sintered with 1 % of Magnesium Oxide (MgO) by weight. The soaking time required for densification of alumina powder with MgO additive to its near theoretical value was determined. Microstructures of the sintered samples were developed and the hardness of the sintered product was evaluated .This study has shown that the MgO is an effective sintering aid for sintering alumina.

Key words: Sintering of Alumina, Characteristics of Alumina, Density of Alumina, Microstructure of Alumina, Soaking time of Alumina with MgO

1 Introduction

Ceramic materials are important class of materials due to, first because they comprise a large and basic industry and, secondly because their properties are critical for many applications.

Alumina which is also known as Aluminum Oxide is an amphoteric oxide of aluminum with the chemical formula Al2O3. Alumina is one of the most versatile of refractory ceramic oxides and finds it's use in a wide range of applications. Among the oxide ceramics, Alumina, Al2O3, is the most commonly used ceramic because of its high hardness, wear resistance, high modulus, inertness, refractoriness and adequate strength. Because of its high-temperature stability and the retention of strength at high temperatures and low cost starting powder, the Alumina is considered as an important engineering ceramic material.

2 The Process

A. Cold Isostatic Process (CIP)

Cold Isostatic Pressing (CIP) is a shape forming process for the consolidation of metal and ceramic powders. For metals, around 100% theoretical density can be achieved, while ceramic powders that are more difficult to compress can reach about 95% of theoretical density after sintering. The CIP uses a liquid media, such as water or an oil or glycol-mixed water, to apply pressure to the powder. The ceramic powder is crammed in a flexible mold made of rubber and an isostatic pressure is applied, after which the powder takes the shape of the mold. The mold also protects against liquid penetrating into the powder.

B. Sintering

Sintering is a heat treatment process in which a large quantity of loose aggregate material is stipulated to a high temperature adequate to cause the loose material to become a compact solid piece. The temperature administered during the sintering process is less than the melting temperature of the material. The purpose of sintering is to impart strength and integrity to the material.

1. During the initial stage particles begin to remain simultaneously and necks rise between the particles. At the completion of this stage, the grain boundaries are established and grain growth begins to occur. Shrinkage is only a few percent.

2. During the intermediate stage grain growth continues. Continuous pore channels are formed along the grain edge. The cross-section of these channels decreases regularly in anticipation of at the end of this stage, at a relative density of about 95%, the channels are pinched.

3. In the final stage closed pores are formed at the

grain boundaries and grain corners. This final stage may lead to an almost completely dense material by removal of these pores, or, alternatively, the pores perhaps missing inside the grains and the boundaries break away from them: the problem of pore grain boundary interaction becomes dominant. Pore growth will occur as well.

3 Experimentation

A. Mixing of Alumina and Magnesia

A 25 g of alumina and 0.25 g of MgO (i.e., 1% of alumina powder) along with alumina balls were taken in a bowl. The bowl was fixed in a High energy planetary ball milling machine for proper mixing. The mill was run for 8 hours with the following conditions. The specifications of the process are:

- Plate speed=200 rpm
- Bowl speed=460 rpm
- Cycle time=20 min
- On time=10 min
- Off time=10 min
- Total no. of cycles=48 cycles

B. Cipping of Alumina Magnesia

After thoroughly mixing of alumina and magnesia powder the powder mixture was taken out and it was subjected to the CIPPING process. A 25 gram alumina +1%MgO (by weight) powder was taken in a rubber mould. The rubber mould was kept in a CIP -ing machine at a pressure of 2K bar. A dwell time of 2 minutes was given to complete the CIP-ing process. After CIP-ing it was observed with the purpose of the alumina powder became a solid specimen due to the compacting process. After completion of CIP-ing process the Alumina samples were dried in air and were cut into small discs of 10 mm thickness.

C. Sintering of pure Alumina

The cut pieces of green alumina specimens were sintered with different soaking temperatures and dwell times. The heating rate for all sintering studies was kept at 18 °C/min, up to those soaking temperatures. After the appropriate soaking period the specimens were furnace cooled.

Below Table 1shows the observations of sintering of pure Alumina.

TABLE 1
OBSERVATIONS OF SINTERING OF PURE ALUMINA

Sample Sl. No	Sintering Temperature °C	Duration (Hours)
1	1500	3
2	1600	3
3	1700	3
4	1700	5
5	1700	7
6	1700	14

D. Sintering of Alumina with Magnesia

Three samples were cut and the green specimens of alumina + 1% MgO (by wt) were sintered at the same temperature of 1700°C (soaking temperature), but the dwell time was varied from 3-7 hrs. The heating rate up to the soaking temperature for all sintering studies was kept at 18 °C/min. After the appropriate soaking period the specimens were furnace cooled. Below Table 2 shows the observations of sintering of Alumina with Magnesia.

TABLE 2
OBSERVATIONS OF SINTERING OF PURE ALUMINA WITH MAGNESIA

Sample Sl. No	Sintering Temperature °C	Duration (Hours)
1	1700	3
2	1700	5
3	1700	7

E. Measurement of Density of Samples

Density measurements of samples were performed using the Archimedes principle. The Archimedes' principle states that the upward buoyant force that is exerted on a body immersed in a fluid, whether submerged fully or partially, is equal to the weight of the fluid that the body displaces and acts in the upward direction at the center of mass of the displaced fluid

4 Results Discussion

A. Study of Temperature on Density

The sintering study on the effect of soaking temperature was studied on pure alumina specimens. The Figure 1 below shows the results. As show in the graph the sintered density increases as the soaking temperature increases. As the soaking temperature increased from 1500°C to 1700°C the sintered density increased from 62% to 74%.

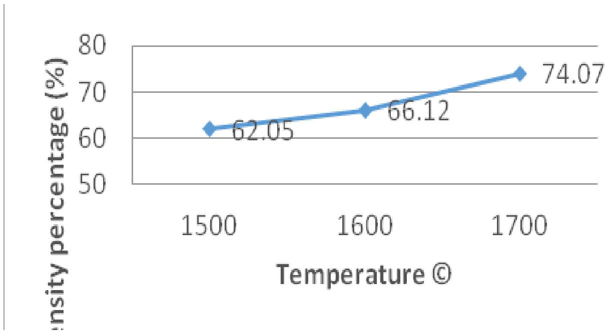

Figure 1: Effect of soaking temperature on density

B. *Studies on Dwell time on Sintering*

The sintering study on the effect of dwell time at various soaking temperatures was done on pure alumina specimens. As shown in the Figure 2, it is found that the sintered density increases as the dwell time increases. As the dwell increased from 3 to 14 hrs the sintered density increased from 74% to 83%.

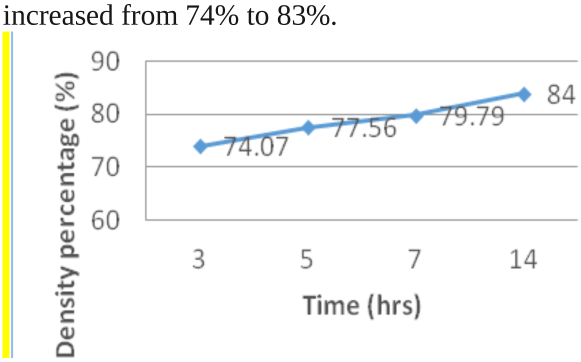

Figure 2: Observations of Sintering of pure Alumina

C. *Study on the effect of Sintering aid*

The effect of sintering aid on the densification of alumina powder was studied. The sintering aid, 1% of Magnesia (MgO) by wt was added to the alumina powder and mixed in a high energy ball mill. All the samples were sintered at a soaking temperature of 1700°C. The dwell time was changed from 3-7 hrs is shown in Figure 3.

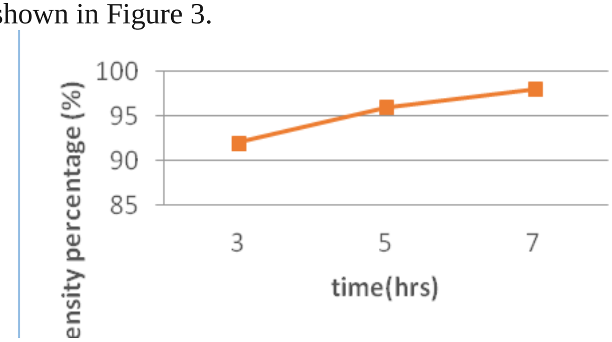

Figure 3: The effect of Sintering aid on density

D. *Study of Micro structure*

Etched microstructure Al_2O_3+ MgO after 3 hours, 5 hours and 7 hours dwell are shown in Figures 4, 5 & 6.

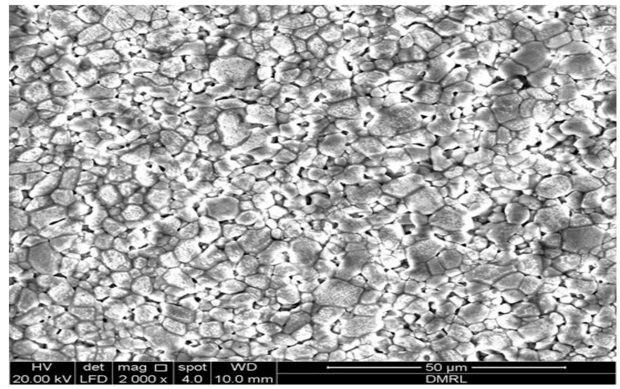

Figure 4: Etched microstructure of Al_2O_3+ MgO after 3 hours dwell

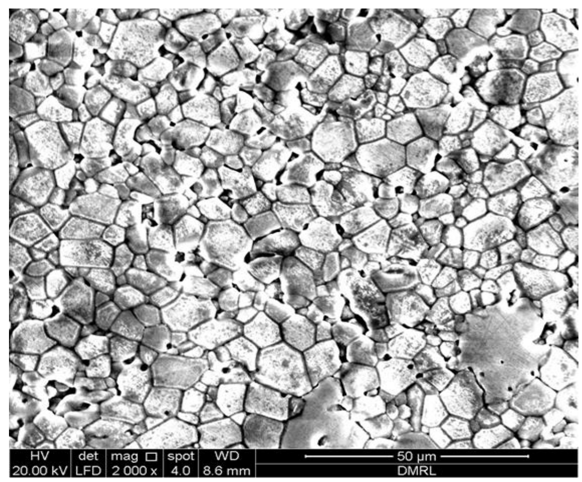

Figure 5: Etched microstructure of Al_2O_3+ MgO after 5 hours dwell

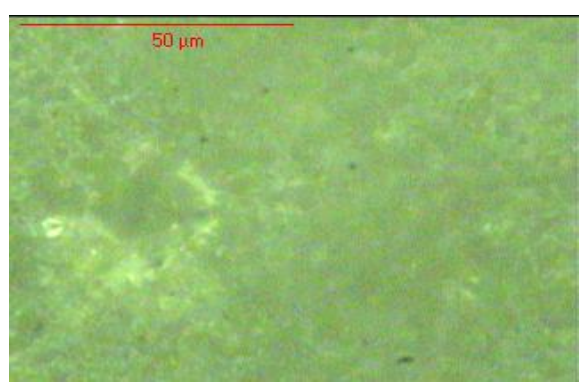

Figure 6: Etched microstructure of Al_2O_3+ MgO after 7 hours dwell

5 Conclusion

In this experiment we have study was done on the Alumina to determine it's sintering characteristics by varying sintering temperature and time. Also the effect of sintering aid MgO on the sintering characteristic of alumina was also studied. The ceramic powder was CIP-ed with 2K bar pressure for 2 min. The CIP–ed specimens were cut into pieces and sintering study at various time and temperature are carried out.

Finally, at 1700°C for 7 hours of dwell time the alumina specimen sintered to its near theoretical density of 98%. It was found that the pure alumina could not be sintered to its fullest value without sintering aid even after sintering at 1700°C for 14 hours. Hence, it was found that the Magnesium oxide is a good additive to sintering alumina ceramic.

References

[1] Erikki Levanen, Tapio Mantyla, "Effect of sintering temperature on functional properties of alumina membranes," *Journal of the European Ceramic Society.*, Volume 22, Issue 5, May 2002, Pages 613-623

[2] C.Legrosa, C.Carry, P.Bowenc, H.Hofmannc "Sintering of a transition alumina: effects of phase transformation, powder characteristics and thermal cycle" *Journal of the European Ceramic Society.*, Volume 19, Issue 11, September 1999, Pages 1967-1978

[3] M.Sathiyakumarab F.D.Gnanama, "Influence of additives on density, microstructure and mechanical properties of alumina" *Journal of Materials Processing Technology:* Volume 133, Issue 3, 20 February 2003, Pages 282-286

[4] J. GurtSantanach, A.Weibel, C.Estournès, Q.Yang, Ch.Laurent, A.Peigney, "Study of parameters, formal sintering analysis and hypotheses on the mechanism(s) involved in densification and grain growth" Journal of Materials Processing Technology: Volume 133, Issue 3, 20 February 2003, Pages 282-286