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Evaluation of Mechanical Properties of Sinterered Nano Alumina Ceramic Powder with Different Doping Concentration

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***Abstract*— Ceramic oxides are the most extensive group of ceramic materials produced today. Ceramics with main component aumina(Al_2O_3) are applied in the field of electro ceramics (insulators) or industrial refractories. In alumina sintering ZnO and MgO were used as doping element. The purpose is to increase the strength of their bond, control their grain size and make them less prone to fracture in the service. In the present work, densification of alumina has been studied in presence of ZnO and MgO. The effect of ZnO was observed in presence of MgO. Four batches of different composition were made. First batch was with pure alumina, 0.1wt% ZnO was added with alumina in the second batch, in the third batch 0.1wt%ZnO and 0.25wt% MgO were added with alumina and in the last batch 0.1wt%ZnO and 0.5wt% MgO were added with alumina. Pellets of specific composition of different batches were sintered at 1400°C, 1450°C and 1500°C and characterized for densification, hardness and wear resistance. Experiments were conducted to determine the appropriate temperature and composition for better physical and mechanical properties. The best combination of all properties has been obtained with 0.1%ZnO-0.25%MgO-99.65%Al_2O_3 composition sintered at 1450°C.**

***Index Terms*— sintering, alumina, temperature, density, hardness, wear resistance, densification, grain size, surface area.**

I. INTRODUCTION

Sintering is a process in which the loose or compacted aggregate of powders are heated below melting temperature of the base oxide with or without the application of external pressure in order to transform it to a more dense material by inter particle bonding [1]. It is a progressive transition without melting, from a state consisting of an agglomeration of metallic particles to a massive state free from porosity and having the desired physical and mechanical characteristics [2].

Sintering is a thermal treatment that bonds particles together into a solid, coherent structure by means of mass transport mechanism occurring largely at the atomic level [3]. It is recognized that the sintering behavior depends on many characteristics of powders and sintering conditions e.g. composition of raw material, particle size, size distribution and shaping methods [4]. Mass movements which occur during sintering consist of the reduction of total porosity by diffusion followed by material transport, mostly density of a collection of grains increases as material flows into voids, causing a decrease in overall size [5]. The initial powder (green body) has a large surface area relative to its volume; this surface area provides the driving force in sintering, which is the reduction of free surface energy resulting from the surface area of the particles [6].

Traditionally, but rather artificially the oxide ceramics are divided into traditional and advanced groups. The traditional ceramics include mostly silica-based products prepared from natural raw materials (clays), including building parts (bricks, tiles), pottery, sanitary ware, and porcelain, but also ceramics with other main components (e.g. alumina, magnesia), which are applied in the field of electro ceramics (insulators), or industrial refractories [7]. From the point of view of the volume of production, polycrystalline alumina is the material most frequently used as ceramics for structural applications. However, in comparison with for example, silicon nitride , where the influence of various additives on microstructure and properties has been well characterized and understood, and despite several decades of lasting research effort, alumina remains a material with unknown factors yet to be revealed.

In alumina sintering the doping element segregate strongly to the grain boundaries in Al_2O_3, block the diffusion of the ions along grain, leading to reduced grain-boundary diffusivity and decreased densification rate [8]. In the undoped Al_2O_3, surface-diffusion-controlled pore drag governs grain growth; in the doped materials, no grain-growth mechanism could be unambiguously identified. Overall the dopant decreases the coarsening rate, relative to the densification rate, and hence, shifted the grain-size-density trajectory to higher density for a given grain size. It is believed that the effect of the additives is linked strongly to their segregation to the Al_2O_3 grain boundaries [9].

As the sintering temperature increases, the rate of grain growth and rate of densification increases. Addition of doping element generally decreases the sintering temperature [10]. Magnesia has a beneficial effect in this respect. Magnesia appreciably decreases the sintering temperature. Generally, sintering temperature for pure alumina without any dopant is above 1600°C, but by adding doping element it can be lowered to 1400°C.

Lower grain size and higher density give better mechanical property. To get a minimum grain size and maximum densification is the aim of the present experiment. But with the increment of temperature this two factors contradict with each other. So an optimum temperature was to be identified to get grain size to density trajectory to a minimum value.

II. EXPERIMENTAL PROCEDURE

2.1 Specification of Raw Materials

Alpha-Aluminum Oxide (Alumina) of purity 99.87%, 100% alpha phase, average particle size 150nm with a specific surface area ~10m²/g was chosen. Magnesium Oxide nano powder of purity 99.9%, particle size ~30nm (BET), with a

specific surface area >50 m^2/g was selected for the experiment. Zinc Oxide nano powder of purity 99.7%, average particle size 30nm with a specific surface area ~35 m^2/g was selected for the experiment.

2.2 Batch Preparation

For the preparation of the slurry an appropriate amount of alumina, magnesia and zinc oxide was weighed out by using an electronic balance. Four Batches of samples were prepared with the following composition shown in Table1.

Table1: Composition of different Batches

Batch	Composition
1	Pure Al_2O_3
2	0.1%ZnO-99.9%Al_2O_3
3	0.1%ZnO-0.25%MgO-99.65%Al_2O_3
4	0.1%ZnO-0.5%MgO-99.4%Al_2O_3

2.3 Mixing

The weighed amount of one batch was taken into a clean pot containing zirconia ball. Then sufficient amount of acetone was added to it, which acts as milling media. The height of acetone in the pot was at least twice than the height of powder or more. Then the pot was subjected to milling action for about 18 to 20 hours. The milling was carried out in a locally made milling machine consisting of a motor and two shafts. The motor speed was 900 rpm and by using variable diameter pulleys around 150 rpm speed was produced in the shafts. The milling was carried out approximately at 150 rpm for about 18 to 20 hours. After milling the pot was taken out and the slurry was extracted from the zirconia balls by means of a sieve. Then this slurry was dried in the oven at a temperature 120°C for 24 hours. After drying few drops of polyvinyl alcohol were added as binder according to the amount of powder. Then the powder was dried for more 20 minutes.

2.4 Cold Pressing

The required amount of powder for desired thickness was weighed for making ceramic cutting tools. This weighed powder was taken in a 13mm diameter die. After assembling it was taken under pressure block. Then pressure was applied on the powder for compaction. The amount of applied pressure was 2.0-2.20 ton for each sample. After 2 minutes pressure was released by releasing pressure valve. Slight backpressure was also applied for removing sample from die. Then the sample was kept on the foil paper.

2.5 Drying

The green samples were dried at a temperature of about 100-110°C for 24 hours in a dryer and stored in a cool dry place.

2.6 Sintering

Dried samples were sintered at different sintering temperatures in an electrically heated furnace. Single stage sintering was performed. First the samples were slowly heated at the rate of 3°C/min to 500°C; hold there for about 1 hour for removing binder and other volatile matter. The polyvinyl alcohol that was used as binder will be completely removed from the samples. During this period it was taken care that the rising of temperature was slow enough to expel the entrapped gas from the product, so that no crack can form. Then samples were heated at the rate of 15°C/min to required sintering temperature, hold at that temperature for 2 hours and then slowly cooled to room temperature. The temperatures used for sintering were1400°C, 1450°C and 1500°C for 2 hours.

III. Determination of Properties

The samples of all the four batches were subjected to hardness test. The %theoretical density and wear resistance were also measured. All the results of hardness test, wear rate and average percentage theoretical density were tabulated in Table2, Table3, and Table4.

3.1 Hardness Test

The hardness of the samples was measured by using Vickers micro hardness testing machine. The indentations were made on the specimens using 1kg load for 30 seconds. The diagonal of the indentations were measured in the SEM.

Table 2: Measured Hardness of all batches sintered at different Temperature

Sintering temperature (°C)	Batch	Vickers hardness (GPa)
1400	1	14.01
	2	16.43
	3	17.41
	4	17.97
1450	1	16.4
	2	17.58
	3	18.29
	4	19.15
1500	1	19.29
	2	20.57
	3	21.34
	4	20.32

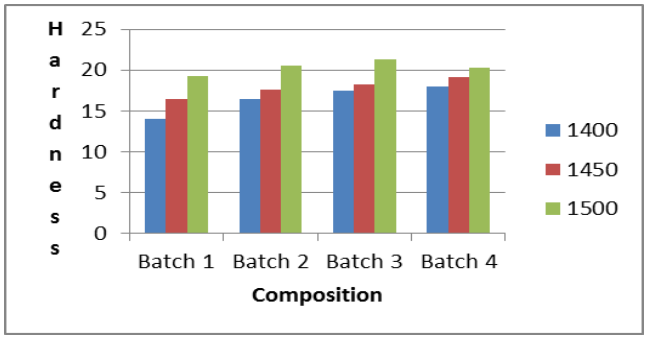

Figure1: Composition versus Hardness at different Temperature

From Figure1 a gradual increase in hardness with increase in sintering temperature was observed for MgO and ZnO stabilized alumina in the temperature range 1400°C to 1500°C. Maximum value of Vickers' hardness was obtained for 0.1%ZnO-0.25%MgO-99.65%Al_2O_3 composition sintered at 1500°C.

3.2 Wear Resistance Measurement

In this project to measure wear property pin on disc method has been used. Wear of samples were measured by using them to operate on the moving discs in the wear testing machine. The discs used were made of hardened steel. The speed of the disc was 1100rpm for each operation. Test time was 30 minutes.

Table 3: Measured Weight Loss of all batches sintered at different Temperature

Sintering temperature (°C)	Batch	Weight loss per minute (gm/min)
1400	1	1.89×10^{-4}
	2	1.76×10^{-4}
	3	1.63×10^{-4}
	4	1.48×10^{-4}
1450	1	1.39×10^{-4}
	2	1.32×10^{-4}
	3	1.15×10^{-4}
	4	0.66×10^{-4}
1500	1	1.46×10^{-4}
	2	1.38×10^{-4}
	3	1.21×10^{-4}
	4	0.75×10^{-4}

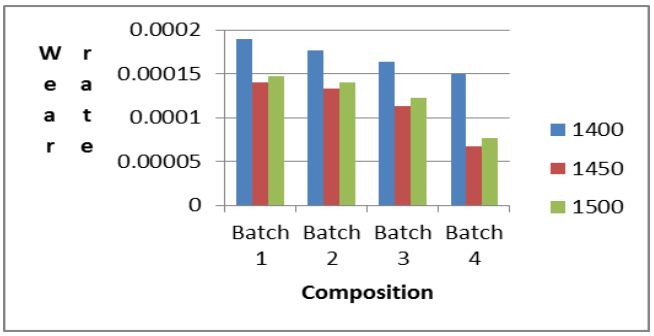

Figure2: Composition versus Wear Rate at different Temperature

From Figure2 it can be seen that for all compositions with increase in sintering temperature wear rate gradually decrease upto 1450°C but then slightly increased at 1500°C. Minimum wear rate was observed for the composition 0.1%ZnO-0.5%MgO-99.4%Al_2O_3 sintered at 1450°C.

3.3 Density Measurement

The % theoretical density (%TD) for all samples of all the four batches was measured.

Table 4: Measured %Theoretical Density of all batches sintered at different Temperature

Sintering temperature (°C)	Batch	%Theoretical density (%TD)
1400	1	90.41
	2	91.35
	3	92.68
	4	92.71
1450	1	91.9
	2	92.41
	3	92
	4	92.34
1500	1	90
	2	92.27
	3	92.39
	4	92.24

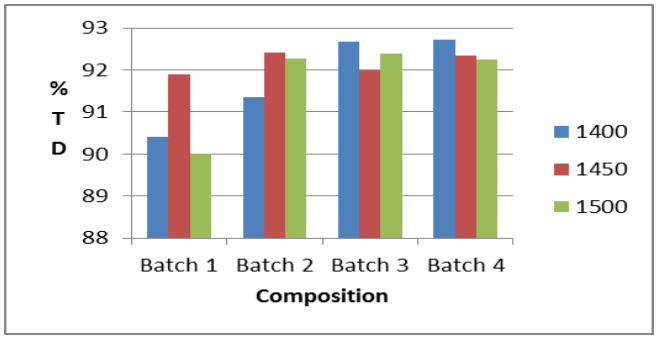

Figure3: Composition versus % theoretical density at different Temperature

From Figure3 it can be seen that for all compositions sintered at 1400°C, with increase in sintering temperature density gradually increases. It is also evident from the chart that if MgO percentage increases, density also increases but with MgO addition and with increasing temperatures simultaneously density decreases. The maximum percentage of theoretical density was found for the alumina ceramic having composition of 0.1%ZnO-0.5%MgO-99.4%Al_2O_3 sintered at 1400°C. But in the case of 0.1%ZnO-0.25%MgO-99.65%Al_2O_3 ceramic %theoretical density is high at 1400°C then density decreases at 1450°C and then increases at1500°C.

IV. Conclusion

From the present work the following conclusion can be drawn:

- The hardness of both ZnO and MgO doped alumina ceramic increases consistently with increasing sintering temperature. Higher percentage of doping concentration retards the grain growth of Al_2O_3 and can provide the pinning effect. The maximum Vickers' hardness value was obtained for 0.1%ZnO-0.25%MgO-99.65%Al_2O_3 composition sintered at 1500°C.
- For all compositions with increase in sintering temperature wear rate gradually decrease up to 1450°C but then slightly increase at 1500°C. The minimum wear rate was shown by 0.1%ZnO-0.5%MgO-99.4%Al_2O_3 sintered at 1450°C.
- With increasing sintering temperature and higher MgO and ZnO content high degree of densification has been achieved. The %theoretical density has gradually increased for alumina ceramic having composition of 0.1%ZnO-99.9%Al_2O_3 and Pure Al_2O_3 up to 1450°C and then decreased slightly at 1500°C due to grain boundary coarsening at high temperature.
- In the case of 0.1%ZnO-0.5%MgO-99.4%Al_2O_3 ceramic the %theoretical density is high at 1400°C, then decreases up to 1500°C where MgO content increases up to 0.5%. So composition 0.1%ZnO-0.25%MgO-99.65%Al_2O_3 sintered

at1450°C is the best in case of %theoretical density.

- Overall, the best combination of all properties has been achieved with composition 0.1%ZnO-0.25%MgO-99.65%Al_2O_3 sintered at 1450°C.

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