

## Effect of Micro-Nano Particle Size Admixture on the Properties of Alumina

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### Abstract

Alumina ceramic is widely used because of its excellent chemical, thermo mechanical and dielectric properties. But low fracture toughness and flexural strength have limited some application of alumina ceramics. Size and distribution of particles have impact bearing on the structure and properties of ceramic materials. In this study, to investigate the effect of particle size of alumina, micron and nano-sized alumina particles were mixed in two different ratios, then pressed and sintered using conventional solid state sintering route. To compare the mechanical properties, 100% micro alumina and 100% nano alumina were also prepared. Sintering time was varied to select appropriate sintering cycle. Physical properties like density, apparent porosity and mechanical properties like Vickers hardness (HV), fracture toughness, at room temperature were evaluated. Microstructure was observed by means of a field emission scanning electron microscope (FESEM). It has been observed that improved properties can be obtained from the micro-nano particle size admixture.

Keywords: particle size, alumina ceramic, micro-nano admixture, sintering.

### 1. Introduction

Ceramics are usually associated with a combination of covalent, ionic and sometimes metallic bonding, having important structural, mechanical, thermal and electronic properties [1]. The structural properties of ceramics depend on the microstructural characteristics e.g., grain size, porosity, the presence of second phase etc., derived from their sintering process [2]. The densification behavior, on the other hand, depends largely on the particle size, particle packing of green compact, heating schedule etc. [3]. In recent years, the fabrication of nano-sized particles has been developed and due to their smaller particle size and higher specific surface areas, densification behavior and properties has improved greatly [4]. Despite their high sinterability, proper sintering of nanoparticles is often hindered by accelerated grain growth [5, 6] and agglomeration tendency [7-10]. If micro and nano-sized particles are mixed, finer particles may fill the interstices between coarser particles and increase the density [11]. Furthermore, to obtain high density, pore free microstructure and clean grain boundaries, appropriate sintering condition is required [12].

In this work, the effect of particle size on the admixture of micro-nano particles of alumina was studied. For this purpose, nano-alumina were mixed with micro-alumina in increasing ratios and 100% micro-alumina and 100% nano-alumina were also prepared to compare the properties of the admixture.

### 2. Experimental procedure

#### 2.1. Raw materials

The raw materials used in this work were two particle sizes of alumina ( $\text{Al}_2\text{O}_3$ ). Locally supplied micro-alumina with average particle size of  $12\mu\text{m}$  (using SEM) and alpha-  $\text{Al}_2\text{O}_3$  nanopowder (containing 99.85% purity) with an average particle size of 150 nm (from BET SSA and SEM) produced by Inframat Advanced Materials were mixed in four different ratios to prepare the ceramics. 0.1 wt.% MgO nanopowder was added in each composition as sintering aid. Four different composition ratios of the admixtures are shown in Table 1.

Table 1. Composition ratios of admixtures

Sample ID	S1	S2	S3	S4-1, S4-2
Micro alumina	100	95	90	0
Nano alumina	0	5	10	100

## 2.2. Sample preparation

The appropriate percentage of raw materials were weighed, dissolved in acetone, and then, taken for milling for about 22 hours to obtain proper homogenization. The milling was carried out in a high density polyethylene (HDPE) pot containing zirconia balls. The slurry prepared from milling was extracted from the zirconia balls and dried in a dryer for about 24 hours at 100°C to evaporate moisture and acetone. The dried sample was crushed to the finest size possible. Liquid binder PVA (polyvinyl alcohol) was then added to the powder and compacted using a uniaxial pressing machine.

The green compacts were dried for 24 hours at about 100°C. After drying, the samples were sintered with proper sintering cycle.

## 2.3. Sintering condition

Mechanical properties of alumina ceramics are influenced by sintering temperature and time [13]. Due to higher sintering stress, nanoparticles show higher densification rate than coarser particles. The densification of these powders is often followed by grain growth [5]. Rapid grain growth occurs at the final-stage of the sintering process [14]. In order to obtain full densification, proper sintering cycle is required. In the present study two different sintering cycles were used. 4 batches of alumina were first sintered at 1500°C for 4 hours with a pre-coarsening stage at 1000°C for 8 hours. But from microstructural observation and obtained mechanical properties, it was found that densification of all compositions was inferior except 100% nano alumina. To improve the densification behavior, sintering time was increased to 8 hours with the pre-sintering time of 12 hrs. To keep the fabricating parameters same, all 4 batches were sintered in this new sintering condition.

## 2.4 Testing method

Green densities of the compacted pellets were determined by measuring diameter and thickness of the pellet using slide callipers. After sintering, true density was measured using Archimedes' principle. Hardness was measured in VICKERS' HARDNESS TESTER by applying 9.8N load for 10 seconds for S1, S2, S3 and 19.6N load on the samples S4-1 and S4-2. Fracture toughness was measured by applying 29.4N load for S1, S2, S3 samples and 49N load for S4-1 and S4-2 samples. Microstructures were observed in Field Emission Scanning Electron Microscope (FESEM, Model: JEOL JSM-7600F) ranging from 2000 to 10,000.

## 3. Result and discussion

### 3.1 Microstructural analysis

Figure 1a shows that elongated grains and pores are observed in 100% micro-alumina due to abnormal grain growth. By mixing 5% and 10% nano-alumina, the abnormal grain growth was hindered and grain refinement was obtained. The average grain size of S2 (95-5) and S3 (90-10) was 0.90µm and 0.77µm respectively. The nano particles have the pinning effect which resulted in grain refinement but due to the agglomeration tendency of nano particles the grain sizes were not homogeneous and porosity was observed.

From the microstructure of the S4-1 sample, i.e., 100% nano alumina (Fig.1e), the average grain size was 1.08µm which is larger than the average grain size of S2 (95-5) and S3 (90-10) samples. For the sample S4-2 the average grain size obtained was 1.21µm (Fig.1d). Abnormal and excessive grain growth occurred due to higher holding time at sintering temperature.

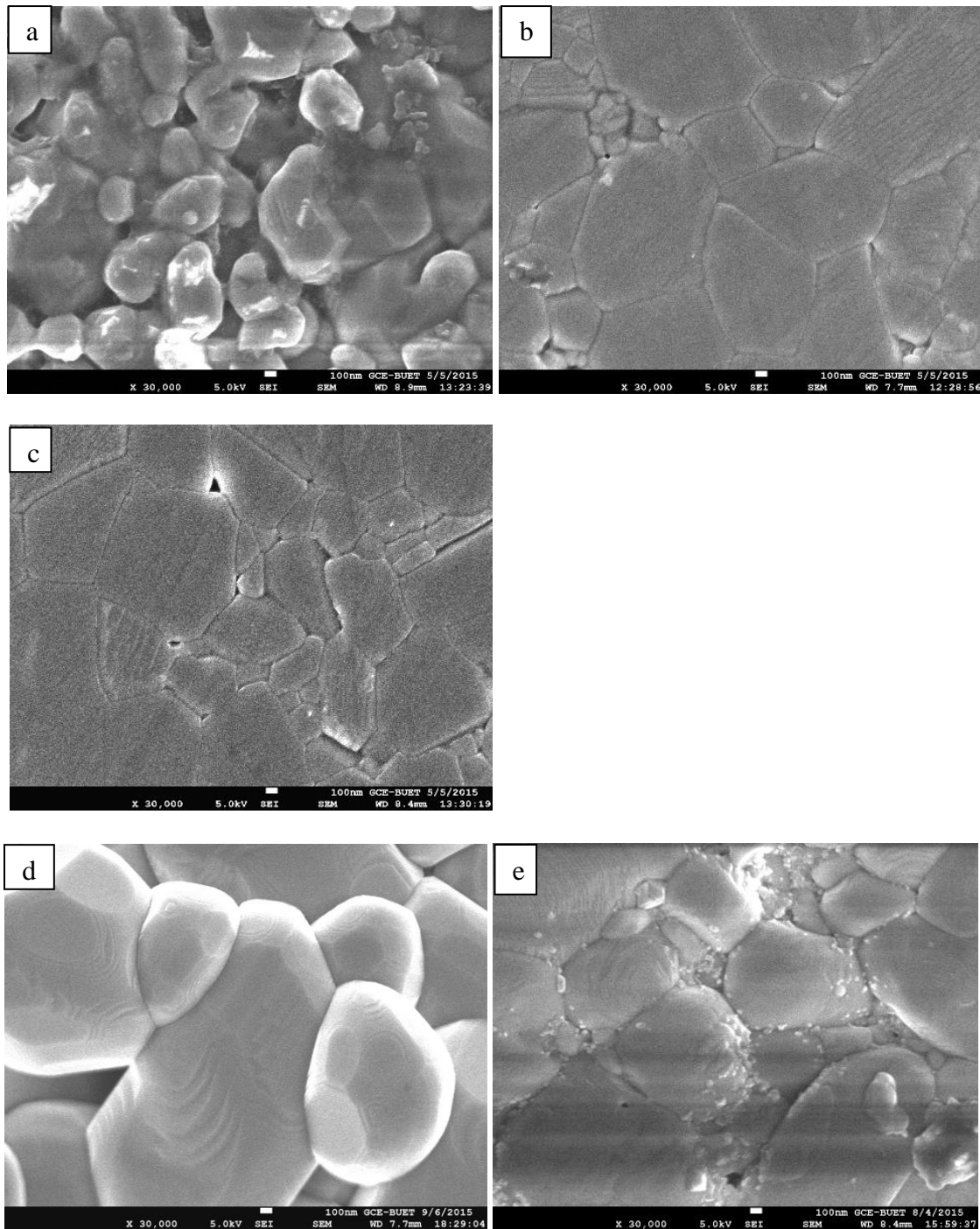


Figure 1. (a) S1 (100:0) (b) S2 (95:5) (c) S3 (90:10) (d) S4-2 (0:100) sintered for 8 hours (e) S4-1 (0:100) sintered for 4 hours, x 30,000.

### 3.2 Comparison of mechanical properties

The fabricating parameters and obtained mechanical properties are listed in Table 2. It can be seen that the properties of the admixture of micro-nano particles increased than the pure micro alumina due to the addition of nano-scale alumina. Nano-scale monolithic alumina obtained the higher properties of all when sintering time was 4 hours .When the sintering time was increased for micro-compositions, all properties of nano-alumina decreased substantially.

Table 2. The fabricating parameters and mechanical properties of different composition of alumina ceramic

Composition	Sintering temperature, °C	Sintering time, hrs	%Theoretical Density	Vickers' Hardness, GPa	Fracture Toughness, MPa.m <sup>1/2</sup>
S1 (100:0)	1500	8	86.9	10.04	3.41
S2 (95:5)	1500	8	94.4	16.18	5.59
S3 (90:10)	1500	8	93.5	14.19	4.84
S4-2 (0:100)	1500	8	96.22	9.77	3.21
S4-1 (0:100)	1500	4	95.5	20.64	5.07

### 3.2.1 Density

For better comparison, variation of sintered density obtained after sintering has been presented in Fig. 2(a) as percent theoretical density. It can be seen that minimum relative density is obtained for 100% micro-alumina because of its coarser particles contributing to highly porous matrix during compaction. After sintering, this porous compact retains significant porosity, though it undergoes high percentage of shrinkage [11]. By mixing 5% nano alumina with 95% micro alumina, density increases significantly because nanoparticles fill the pores of the micro alumina matrix [13]. Again, by increasing nanoparticles to 10%, density of the matrix decreases. This can be related to the agglomeration tendency of the nano-sized particles which causes to retain pores in the agglomerated zones. As a result density tends to decrease [15].

Due to the higher surface activity of nanoparticles, 100% nano-alumina shows higher sintered density than purely micro alumina or micro-nano admixture [17]. For higher sintering time, densification improved but grain size increased abnormally (Fig.1d).

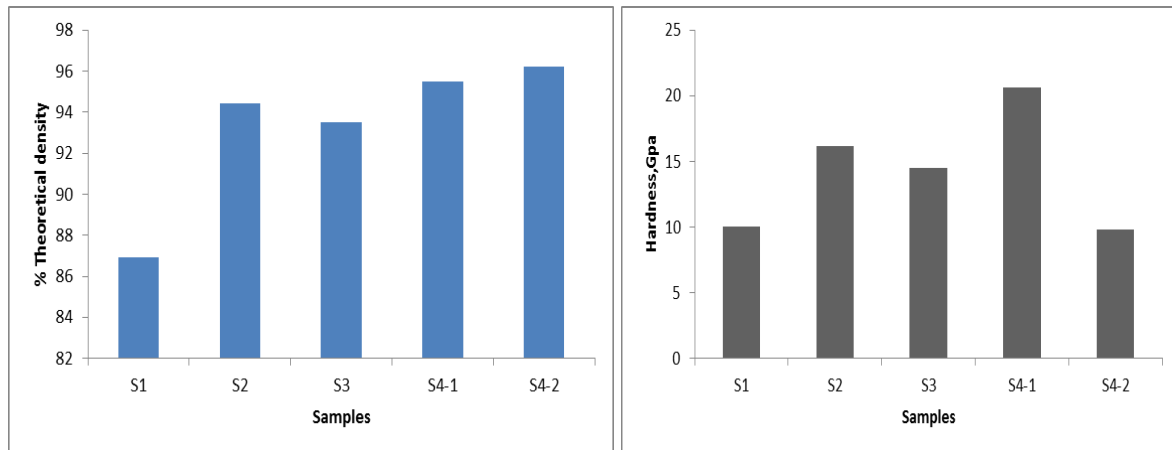


Figure 2. (a) Variation of sintered density (b) variation in hardness [S4-1 and S4-2 denoting 100% nano-alumina sintered for 4 hours and 8 hours respectively]

### 3.2.2 Hardness

The effect of particle size on hardness value is same and can be explained in the same way as that on density variation. By mixing 5% and 10% nano alumina increased hardness than 100% micro alumina due to dispersion of nanoparticles in the micro alumina matrix. For S3 (90-10), hardness decreased than S2 (95-5). This can be attributed to the agglomeration tendency when nanoparticles increase in amount [15].

On the other hand, 100% nano-alumina obtained highest hardness for lower sintering time. Nano particles are much closer to each other than micro-sized particles, which can strongly resist indentation and result in high hardness [16]. With increasing sintering time, due to abnormal grain growth [17], hardness of 100% nano-alumina decreased to a very low value, even lower than that of 100% micro-alumina.

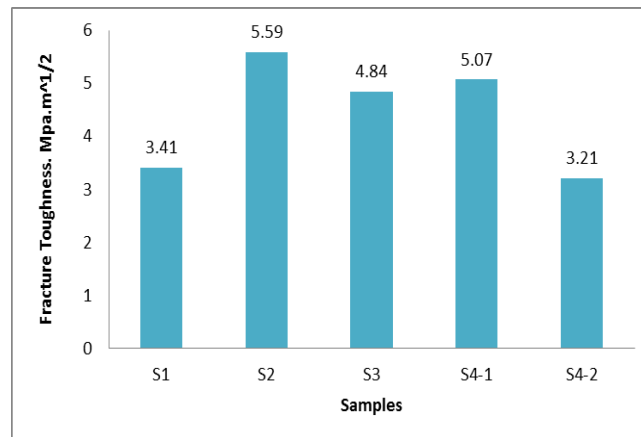


Figure 3. Variation in fracture toughness [S4-1 and S4-2 denoting 100% nano-alumina sintered for 4 hours and 8 hours respectively]

### 3.2.3 Fracture toughness

As a brittle material, micro alumina has low fracture toughness, which is consistent with the result of our present work. With addition of 5% nanoparticles, fracture toughness increased due to high fracture energy dissipation during crack growth. This may be attributed to the reinforcement of grain boundary by nanoparticles, which results in the transition from intergranular to transgranular fracture mode [17]. With 10% addition of nano-alumina, fracture toughness decreased than the 95-5 mixture. This may be related to the grain size refinement, as from Fig.1, grain size of 90-10 mixture was smaller than all other compositions [18].

For 100% nano alumina fracture toughness was moderately high for lower sintering time. With increased sintering time fracture toughness deteriorated significantly. Due to abnormal grain growth, mechanical properties decrease [17]. This may reduce fracture toughness as well.

## 4. Conclusion

To study the effect of particle size, micro- and nano-alumina were mixed in two different ratios and to compare, 100% micro- and 100% nano-alumina were also prepared. Sintering time was varied to obtain better mechanical properties. For lower holding time at sintering temperature, higher properties were obtained for 100% nano-alumina, which deteriorated significantly for increased sintering time. On the other hand, for higher holding time, properties of S2 (95-5) improved greatly that it can be compared with the higher values of 100% nano-alumina. For S3 (90-10) density, hardness, fracture toughness decreased than S2 (95-5), but were higher than that of 100% micro-alumina.

## 5. References

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