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Alumina-based Ceramic Material for High-voltage Ceramic Substrate

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ABSTRACT

The paper presents the study of the particle size distribution, surface area and their effect on sintering of alumina (Al_2O_3) using additives such as magnesium oxide (MgO) and silica (SiO_2), so that the samples could be sintered to high relative density (~ 97.43 %) with controlled grain growth. However, the use of MgO along with SiO_2 on Al_2O_3 produced the powder compacts having high Green density, sintered density with minimum porosity to achieve high dielectric strength ceramic material, so that material can be used for high-voltage insulator applications.

Keywords: Alumina, silica, magnesium oxide, particle size, surface area, Green density, sintered density, dielectric strength, dielectric constant

1. INTRODUCTION

The ceramic processing is complicated and influenced by several factors, the intrinsic properties are the characteristics of raw powders such as powder purity, particle size distribution, surface area, etc and influence of additives have profound effect on sintering of alumina (Al_2O_3) to a required sintered density with controlled grain size and porosity. It is also reported by Yeh and Sacks[1] that the microstructure of a powder compact has a tremendous effect on the sintering behaviour. There are a variety of characteristics which determine the nature of microstructures, including particle size and particle packing.

The importance of preparing powders and powder compacts has been emphasised in several recent studies of sintering. However, model investigations concerning the effect of particle size distribution and particle packing on sintering behaviour are still limited. It is also suggested by Occhionero[2] that the shrinkage behaviour depends upon surface area and particle size distribution. The higher surface area powder exhibits sintering shrinkages at lower temperatures. During the processing, the influence of small amount of additives (dopants) ie magnesium oxide (MgO) on sintering process has been widely studied by several scientists[3-6]. Basically, additives enhance the sinterability.

A common goal of those studies is to obtain desirable sintering of alumina to theoretical density with microstructure of a powder compact having controlled grain size and minimum porosity. However, no work was carried out on the effect of MgO along with silica (SiO_2) on sintering of alumina to the required density. The purpose of this study is to investigate the effect of particle size, particle size distribution, surface area, and influence of additives (MgO and SiO_2) to obtain the desired theoretical density of alumina with high dielectric strength, controlled grain size distribution, low porosity so that the material can be subsequently used for

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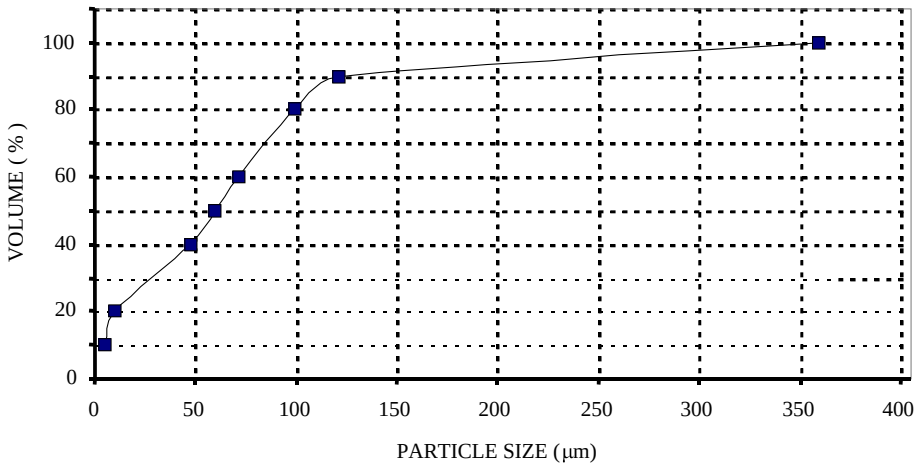

Figure 1. Particle size distribution of raw (unmilled) alumina powder

fabrication of critical-shape alumina-ceramic substrate for high-voltage insulator applications for mounting electronic chip circuit.

2. EXPERIMENTAL PROCEDURE

2.1 Synthesis of Material

Raw materials, viz., Al_2O_3 (Indal), *MgO* (J.T. Baker, UK), and SiO_2 (Qualigens) with purities of minimum 99 per cent were obtained and used as starting materials. For the synthesis of material, the following steps were involved:

Step 1. *Particle Size Reduction*

The alumina powder having an average particle size, 59.6 μm (as shown in Fig. 1) was found to be very coarse with broad particle size distribution. The powder was milled in rubber-lined jars with distilled water as medium and cylindrical alumina grinding media (12.5 mm x 12.5 mm). The milling was carried out from 24 h to 48 h. to obtain an average particle size up to 3μm with narrow particle size distribution as shown in Fig. 2.

Step 2. *Modification by Additives*

The alumina powder with the desired particle size was taken for doping and processing. Two powders were processed for experimental purpose. In the first powder, *MgO* (0.05 to 1.0 Wt %) along with SiO_2 (0.05 to 1.0 Wt %) was added in the form of crystalline gel as material type 1 and the other powder was doped with *MgO* (0.05 to 1.0 Wt %) as material type 2. Both the powders were mixed and milled for homogenous mixing.

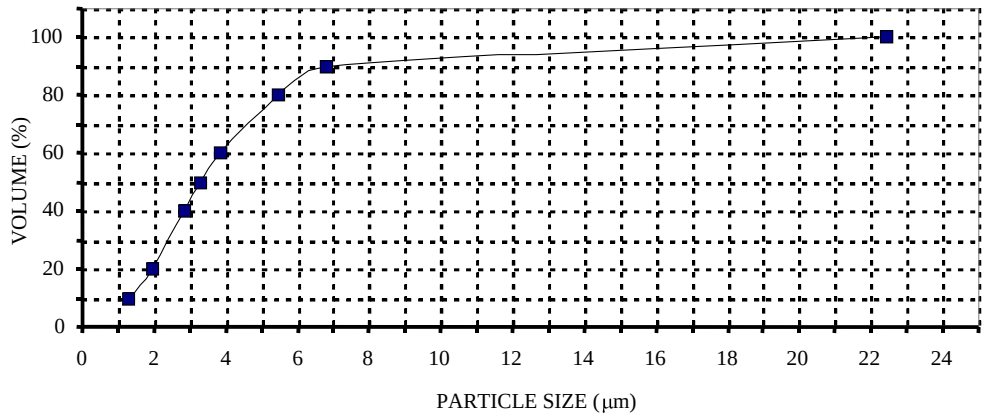

Figure 2. Particle size distribution of milled alumina powder

Step 3. *Calcination*

The mixed alumina powder was calcined at 1200 °C to 1400 °C for 4 h to 8 h for completion of the reaction and conversion of γ-alumina to α-alumina[7-9].

Step 4. *Fine Grinding*

Both the reacted alumina powders (*MgO*-doped and *MgO* + SiO_2-doped) were again wet-milled from 24 h to 48 h to obtain the optimum particle size. The particle size obtained for *MgO* + SiO_2-doped alumina was 1.68 μm, and for *MgO*-doped alumina was 1.70 μm.

Step 5. *Binder Addition & Granulation*

The doped alumina powders were granulated by (3 to 5 Wt %) polyvinyl alcohol as binder[10-13]. These granulated powders were used for pressing specimens.

Step 6. *Pressing*

The standard specimens having 20 mm (diameter) and 1.0 mm (thick) discs were pressed on hydraulic press at 136 MPa to obtain the optimum Green density[14-17].

Step 7. *Binder Removal*

The pressed specimens were pre-sintered at 1100 °C for 2 h to 4 h with 2 °C/min to 4 °C /min heating rate.

Step 8. *Sintering*

The sintering was carried out at 1600 °C, 1620 °C, 1650 °C for 10 min, 45 min, 90 min, respectively at heating rate of 3 °C/min to 5 °C/min. The percentage of shrinkage, grain growth, porosity, density was obtained at various sintering temperatures to achieve the optimum grain size and sintered density.

2.2 Characterisation of Material

Testing of Alumina Ceramics

The standard specimens obtained from *MgO*-doped alumina and *MgO* + SiO_2-doped alumina were used to study the various properties and also their properties were compared with the properties reported by Buchanan[18]. The particle sizes of milled alumina powders were measured using particle size analyser, model No. 2000 MU(A), make Malvern, UK. The Green density of the test specimens (compacts) was evaluated by geometric method and was 58 per cent to 60 per cent of the theoretical density as 3.98 g/cc as reported[2-6]. The density of the sintered specimens was determined by Archimedes method (liquid displacement with deionized water). The instrument used was densometer, Contech, model No. CB120. The shrinkage percentage of the specimens (compacts) after heating was measured geometrically.

The sintered specimens were sliced to required sizes for microstructural analysis. The specimens were photographed at several positions using scanning electron microscope (Phillips make) model No. XL30 at 5000X (magnification) and 40 Å resolution. The working distance was 20 mm and accelerating voltage was 25 kV. The bar length shown in the microphotograph is 5 μm = 17 μm. The microstructure of the surface of sintered specimens was tested and it indicated grain growth, grain boundary, grain structure, and influence of additives such as *MgO* and SiO_2 in achieving the required sintered density. The average grain size and porosity were measured using linear intercept method[18-19].

The dielectric strength and dielectric constant of the specimens were measured. Because the dielectric strength values are very sensitive, testing was carried out at room temperature. The high dc electric field (kV/mm) was applied and optimum field taken at insulation breakdown was measured. The dielectric constant of the specimens were calculated wrt the values of the capacitance.

The modified alumina powders were further used to fabricate the ceramic substrate having high dielectric strength required for high-voltage insulator for mounting electronic chip circuit. The critical shape of ceramic substrate having dimensions 6.1 mm (diameter) x 3.0 mm (thickness) were pressed at 56 MPa, sintered from 1600 °C to 1650 °C for 2 h to 4 h to obtain the required shrinkage and dimensions within the tolerance limits.

3 RESULTS & DISCUSSION

3.1 Particle Size, Particle Size distribution & Surface Area

The starting (unmilled) particle size of alumina powder is very coarse with broad particle size distribution from 5.4 μm (10 %) to 120 μm (90 %) with average particle size being 59.6 μm at (50 %) as shown in Fig. 1. The powder is further milled to reduce the average particle size to 3.3 μm as shown in Fig. 2. The particle size distribution of $MgO + SiO_2$-doped alumina is from 0.77 μm (10 %) to 3.38 μm (90 %) with average particle size ~1.68 μm (50 %). While the particle size distribution of *MgO*-doped alumina is from 0.72 μm (10 %) to 3.61 μm (90 %) with average particle size ~1.7 mm, it is seen that the difference in particle size distribution between both the powders is very less. However, it is reported[4,20,21] that both the particle size and particle size distribution have great influence on achieving the required Green density, sintered density, uniform grain size (μm) and no abnormal grain growth.

It has been reported[1] that the densification rates may be enhanced if the particles are very fine (submicron) sized particles. It is due to large driving force and shorter diffusion distances. However, another problem with fine particles is their tendency to form aggregates. So compacts with very fine particles tend to have low Green density, inhomogeneous packing, and large interagglomerated pores. Now in contrast, some researchers have reported[5,22,23] that the particle size distribution (if these have narrow size distribution or broad size distribution), does not affect the sintered density at similiar sintering temperatures, however, the sintered density is affected by the additives. The surface area data is given in Table 1. It is observed that the surface area of unmilled alumina is very small (~ 0.16 m^2/g), while after milling, *MgO*-doped alumina indicates increase in specific surface area (up to 4.25 m^2/g). On the other hand, $MgO + SiO_2$-doped alumina indicates maximum surface area (~ 4.35 m^2/g), which suggests increase of surface area to achieve optimum shrinkage and sintered density.

3.2 Green Density, Sintered Density & Shrinkage Characteristics

The Green density is one of the most important step. It indicates the degree of compaction (formation). From the data given in Table 1, it is infered that $MgO + SiO_2$-doped alumina has high Green density (~ 2.28 g/cc) suggesting better compactability in contrast to *MgO*-doped alumina (~ 2.10 g/cc). The shrinkage during sintering depends on Green density as indicated by the data. However it is seen that specimens with $MgO + SiO_2$-doped alumina showed tremendous increase in shrinkage (~ 15.58 %) with increase in sintering temperature. On the other hand, the specimens of alumina-doped with *MgO* showed ~ 13.37 per cent (max) shrinkage, indicating low sintered density.

However, it is reported by Reed[24] and Chappell[25] that the compacts with low Green density show larger sintering shrinkage, but the results obtained indicate higher shrinkage due to the influence of additives. The final shrinkage depends not only on the Green density but also depends on surface area and the number of pores and their elimination during sintering. The powders with higher surface area exhibit higher sintering shrinkage. The amounts of shrinkages given in the data satisfies the present

Table 1. Green density, sintered density, shrinkage characteristics of $MgO+SiO_2$ and *MgO*-doped alumina specimens

Sample specification	Average Green density (g/cc)	Average sintered density (g/cc) sintering temperature (°C/min.)			Shrinkage (%) at various temperatures (°C/min)			Sintered density(%) at various temperatures (°C/min)		
		1600/10	1620/90	1650/90	1600/10	1620/90	1650/90	1600/10	1620/90	1650/90
$MgO+SiO_2$ -doped alumina	2.28	2.97	3.56	3.80	12.18	14.60	15.58	76.15	91.28	97.43
MgO-doped alumina	2.10	2.77	3.18	3.26	11.36	13.04	13.37	71.02	81.53	83.58

observation. The data given in Table 1 indicates that sintered density increases with sintering temperature, which results in increase in shrinkage (percentage).The density of *MgO* + SiO_2-doped alumina specimens shows increase from 2.97 g/cc to 3.80 g/cc ~ 97.43 per cent with increase in sintering temperature from 1600 °C to 1650 °C, corresponding to 3.98 g/cc theoretical density given. On the other hand, the sintered density of specimen with *MgO*-doped alumina showed an increase from 2.77 g/cc to 3.26 g/cc ~ 83.58 per cent which is comparatively less to *MgO* + SiO_2-doped alumina specimens having comparatively same average particle size of the powder. This indicates the importance of the additives. Further, It is reported in general that if the Green density is on higher side, the sintered density will also be on the higher side. Hence, the importance of sintered density with uniform grain size was reported by several scientists.

3.3 Microstructure Development

3.3.1 *Influence of MgO and SiO_2-dopants on Grain Growth, Grain Size, Porosity Elimination*

The data on microstructure of sintered specimens is given in Table 2 and microphotographs are represented in Figs 3-8. The data indicates an increase in grain size with increase in sintering temperature. The specimens with *MgO* + SiO_2-doped alumina showed controlled grain growth with increase in grain size from 1.82 μm to 2.66 μm from 1600 °C to 1650 °C, while the grain growth observed in case of *MgO*- doped alumina specimens was found to be less uniform, [from 1.51 μm to 2.43 μm (max)]. However, it has been reported by several researchers that the influence of *MgO* on alumina is more pronounced and many of the researchers emphasised its importance in ceramic processing[26-30]. However, very few studied the influence of *MgO* along with SiO_2, but the results of *MgO* + SiO_2-doped alumina suggests better performance than *MgO*-doped alumina during sintering. This is verified by the results obtained.

The microphotographs shown in Figs. 3 to 5 indicate the specimens of alumina doped with *MgO* + SiO_2 and their uniform distribution of grain size with increase in sintering temperature. Fig. 5 shows the specimens sintered at 1650 °C for 90 min showed maximum grain size (~ 2.66 μm) with maximum sintered density ~ 97.43 per cent and minimum porosity ~ 2.57 per cent. However, it is clearly seen from Fig. 8 that the distribution of grain size is nonuniform with little influence of *MgO*-doped alumina as compared to *MgO* + SiO_2-doped alumina, as shown in Fig. 5. The material doped with *MgO* showed maximum grain size (2.43 μm) with porosity ~ 16.42 per cent indicating the presence of porosity in the material. The final grain size of *MgO* + SiO_2- doped alumina sample ~2.66 μm as shown in Fig. 5 is predicted to be larger than that of *MgO*-doped alumina sample of grain size ~ 2.43 μm for the same sintering temperature and time.

The results obtained are somewhat surprising but these are the experimental observations. The microphotograph of Fig. 8 shows the structure of *MgO*-doped specimens with the pores inside and in between the grains, and this has perhaps occurred due to discontinuous grain growth and grain boundary movement. The microstructure of alumina specimens doped with *MgO* + SiO_2 shown in Fig. 5 indicates that the compacts are substantially pore-free, indicating *MgO* + SiO_2-doped helped in reducing grain

Table 2. Average grain size and porosity of *MgO* + SiO_2-doped and *MgO*-doped alumina specimens

Sample specification	Average grain size (μm) at various sintering temperatures (°C/min)			Porosity (%) at various temperatures (°C/min)		
	1600/10	1620/90	1650/90	1600/10	1620/90	1650/90
MgO + SiO_2-doped alumina	1.83	1.89	2.66	23.85	8.72	2.57
MgO-doped alumina	1.51	1.81	2.43	28.98	18.47	16.42

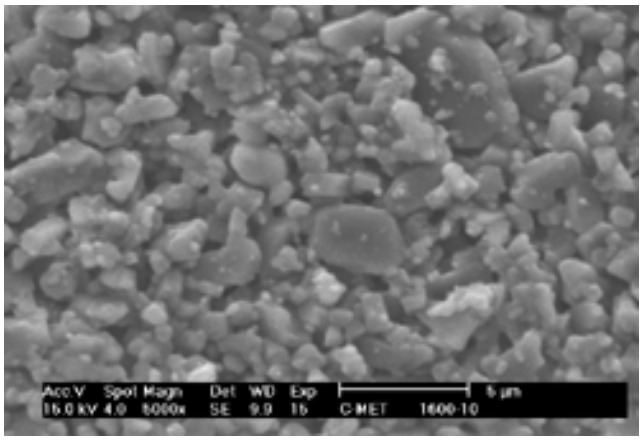

Figure 3. Microphotograph of *MgO* + SiO_2-doped alumina sintered at 1600 °C/10 min.

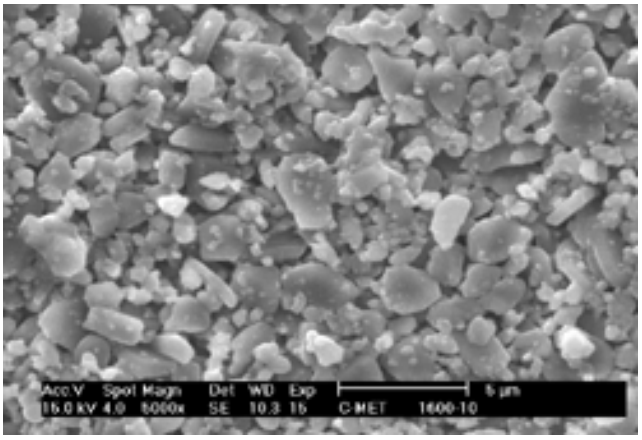

Figure 6. Microphotograph of *MgO*-doped alumina sintered at 1610 °C/10 min.

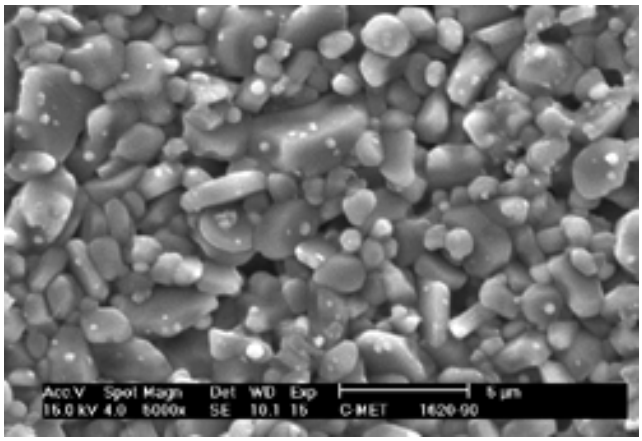

Figure 4. Microphotograph of *MgO* + SiO_2-doped alumina sintered at 1620 °C/90 min.

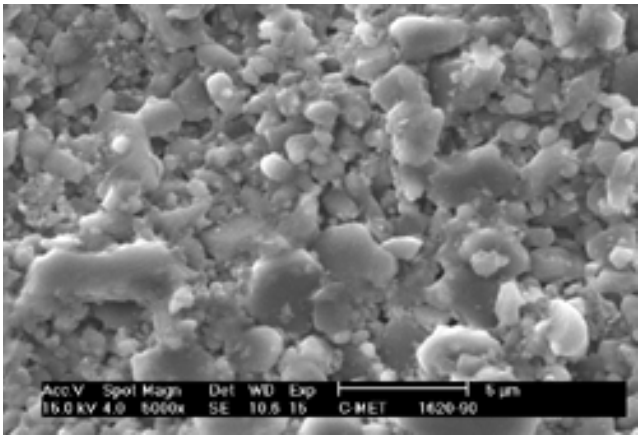

Figure 7. Microphotograph of *MgO*-doped alumina sintered at 1620 °C/90 min.

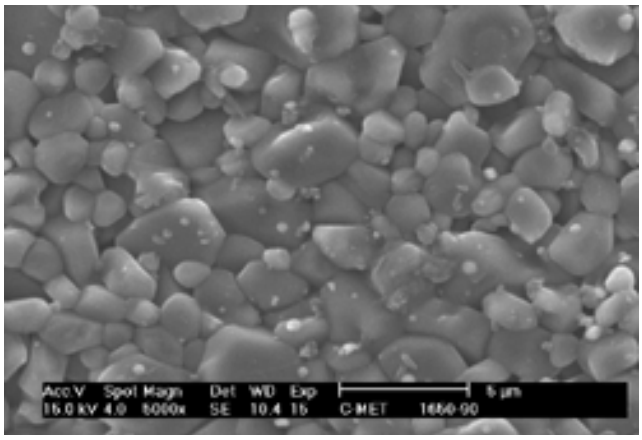

Figure 5. Microphotograph of *MgO* + SiO_2-doped alumina sintered at 1650 °C/90 min.

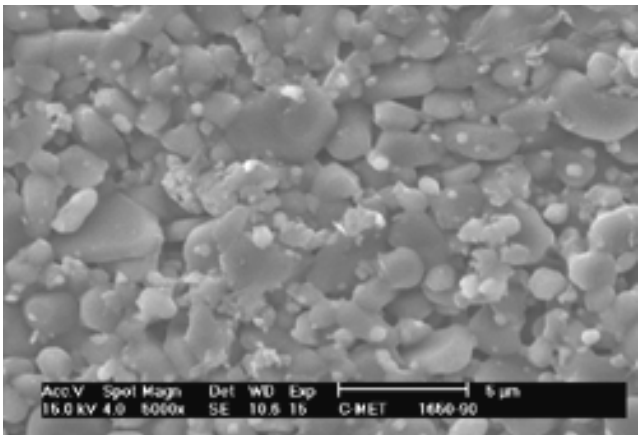

Figure 8. Microphotograph of *MgO*-doped alumina sintered at 1650 °C/90 min.

boundary mobility, and hence, prevented the break away of grain boundaries from pores, which could have initiated exaggerated grain growth.

It has been reported in literature[6,31-38] that dopants are usually added to improve the densification and with the selection of some dopants, near-theoretical density can be achieved with enhancement in grain boundary diffusivity at the expense of surface diffusivity and vapour transport. The suppression of exaggerated grain growth in alumina is due to the effect of dopant such that there is increase in surface diffusivity and pore mobility so that pores remain on grain boundaries until these are eliminated.

Finally, Clarke[39] reported that even after enormous work on *MgO* doping on alumina, the detailed understanding of atomic mechanism through which *MgO* solute acts as grain growth inhibitor in alumina, is not clear. This is due to the lack of understanding of the fundamental grain growth mechanism in ionic solids. However, the chemical characterisation of *MgO*-doped alumina remains one of the obstacle in the complete understanding of the role of *MgO*. Hence, in general, it is observed that with increase in sintering temperature, the grain size increases with increase in sintered density and reduction in porosity, as observed in the specimens of alumina-doped with *MgO* + SiO_2, which is found to be better in processing.

3.4 Dielectric Strength & Dielectric Constant

The data of dielectric strength and dielectric constant is given in Table 3. It is evident from the data that the dielectric strength of the specimens of alumina (*MgO* + SiO_2-doped) increased from 9.7 kV/mm to 28.30 kV/mm with increase in sintering temperature from1600 °C to 1650 °C. On the other hand, the increase in dielectric strength of *MgO*-doped alumina was from 3.37 kV/mm to 16.87 kV/mm, was marginally low. The grain size increased with increase in sintering temperature and sintered density, which affected the increase in dielectric strength, as shown in Figs 9 and 10. However, the dielectric strength decreased with increase in porosity (%), and the dielectric constant of *MgO* + SiO_2-doped alumina was 11.08 (max) in contrast to *MgO*-doped alumina, which was 8.43 (max) at the same sintering temperature of 1650 °C/90 min. This was because of increase in capacitance (pF) value of the material with corresponding increase in sintering temperature.

However, the results obtained wrt dielectric strength and dielectric constant were on the higher side as compared to the results obtained by Buchanan[40-42], *et al*. where they have reported the factors which affect dielectric strength are the microstructural features such as porosity, grain size, cracks, flaws, etc.

The SEM microphotographs clearly show that *MgO* + SiO_2-doped alumina indicates uniform grain size ~ 2.66 μm with less amount of porosity (~ 2.7 per cent),indicating high dielectric strength with high dielectric constant. On the other hand, the results of *MgO*-doped alumina showed ~ 2.43 μm grain size and 16.42 per cent porosity, which indicates low dielectric strength with low dielectric constant. The data obtained in general suggests that the uniform grain size and sintered density result in higher dielectric strength and dielectric constant, which is confirmed by the results obtained. The material with high dielectric strength can be used as insulation material for high electric fields.

Table 3. Dielectric strength and dielectric constant of *MgO* + SiO_2-doped and *MgO*-doped alumina specimens

Sample specification	Dielectric strength (kV/mm) at various sintering temperatures (°C/min)			Dielectric constant (KT_3) at various sintering temperatures (°C/min)		
	1600/10	1620/90	1650/90	1600/10	1620/90	1650/90
MgO + SiO_2-doped alumina	9.70	14.0	28.30	5.32	9.47	11.08
MgO-doped alumina	3.37	9.43	16.87	3.22	4.70	8.43

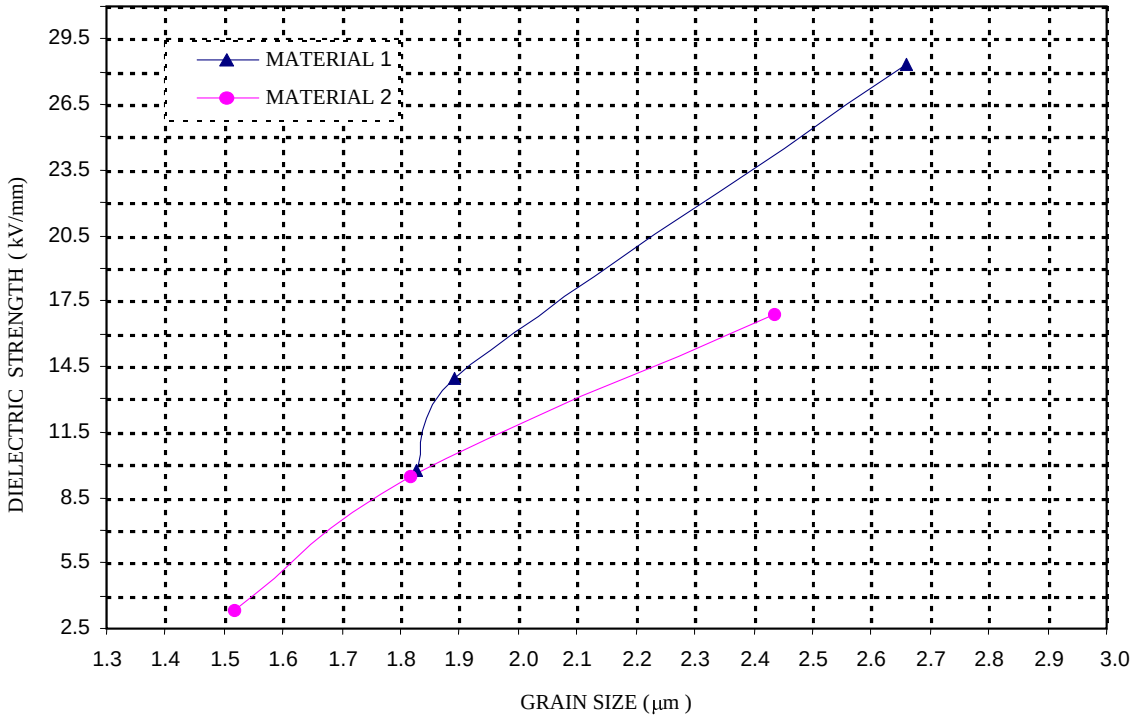

Figure 9. Grain size versus dielectric strength

3.5 Fabrication of Ceramic Substrate

The alumina-ceramics doped with both $MgO + SiO_2$ (material type 1) and *MgO* (material type 2) were used for the fabrication of critical-shape ceramic substrates. The ceramic substrate pressed at constant load and sintered from 1600 °C to 1650 °C indicated maximum sintered density at 1650 °C for material type 1 as shown in Table 1, which is far more superior than material type 2. The dimensions obtained are within the tolerance limit (± 0.1 %). Hence, this suggests the suitability of these materials for high-voltage insulator applications.

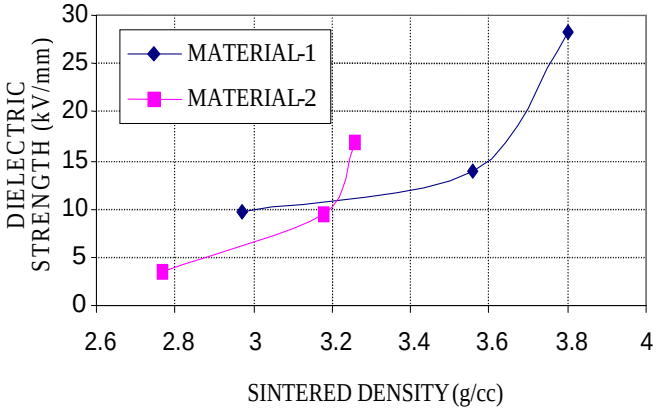

Figure 10. Sintered density versus dielectric strength

4. CONCLUSION

A critical analysis of the data generated suggests that alumina-doped $MgO + SiO_2$ has tremendous influence to achieved required homogeneous compacts (specimens) with optimum Green density, sintered density, minimum porosity, with controlled grain growth and grain size to obtain material with high dielectric strength and dielectric constant, but low conductivity which is found to be more beneficial than alumina-doped *MgO*. The material developed was suitable for fabrication of ceramic substrate for use in high-voltage insulator applications.

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