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Effect of MgO Additive on Microstructure of Al_2O_3

Zawati Harun[1, a], Nur Farhani Ismail[2, b], Nur Azam Badarulzaman[3, c]

[1, 2, 3] Department of Materials and Design, Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, Malaysia

[a] zawati@uthm.edu.my, [b] far_hani11@yahoo.com, [c] azam@uthm.edu.my

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Abstract. In this work, the consolidation of different alumina particles via sintering process was conducted to the compacted alumina pellets. This consolidation was also assisted with the sintering aid, MgO to densify the final ceramic structure. Comparison between the influence of additive to the different particles size of compacted alumina by observing the microstructure and physical properties was conducted. The value physical properties and microstructure clearly show that for both particle size of alumina, MgO additive can increase the density value and improve microstructure properties.

Introduction

Consolidation is one of the most important and crucial part in fabricating ceramic component or structure. In fact, most of the ceramic component that fabricated via plastic forming and powder route for the variety applications must undergo this sintering steps [1]. Basically, this stage is ranging from green body forming with starting from the loose particles to the thermal consolidation or sintering stage [1-5]. Sintering is a process where the fully densification will takes place and involves the development of new ceramic structure [1,6]. Typically, this process need to be carried out at nearly melting temperature of ceramic in order to make sure the diffusion mechanism of compacted loose particles will occur [7]. This mechanism of sintering occurred by diffusion transport of matter along definite path which commonly occur at the boundary of stacked or closed particle as shown in Fig.1 below. The diffusion process that involves during sintering will lead to the interaction between pores and grain boundaries and finally leads to movement of grain growth and the development of a new dense grain structure which preferably in the polycrystalline form [1,8]. Hence, this will increase the strength of the sintered powder due to the bonding and growth of necks between the particles [1-3]. Basically at this stage also, the particles are coalesced (including grain and pore growth) by solid state diffusion with at the same time both of the grain growth and densification occur simultaneously [1,7,9-11]. This elaborated in detail by the previous studies that showed a clear pore growth accompanied by the grain growth development with the existence of high sintering temperature [11,12].

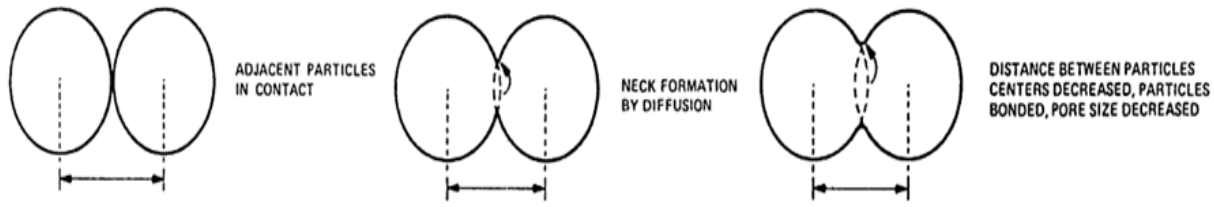

Fig.1. Schematic of Solid State Material Transport by Diffusion [5].

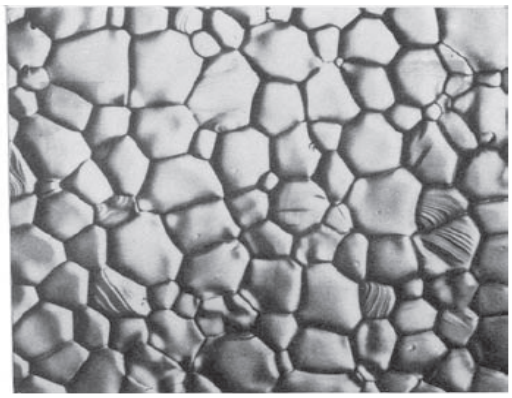

Fig.2. The surface of an Al_2O_3 ceramic during sintering [2,3].

Uncontrolled grain growth is a severe problem that leads to the production of ceramic with undesirable properties such as low strength and crack. Thus, the control of the grain growth during sintering stage is one of the most crucial steps in the formation of good ceramic structure. As this grain growth is directly controlled by the final grain size and grain structure formation, thus the achievement of the desired properties can be achieved by increasing the diffusion distance for matter transport and hence reducing the rate of grain growth. In ceramic materials, grain growth can be described by normal and abnormal grain growth [1-3]. So, the controlling the abnormal grain growth is important in achieving a homogenous and higher density. Basically, the fabrication of the ceramic with high density and controlled grain size is depending on reducing grain growth rate and increasing densification rate or some combination of these two. Some modifications of the fabrication approaches that satisfy one or both these condition include the use of additive (dopant) in order get the desired [7,8,13]. In fact, the use of additive offers a very effective approach for fabricating a presureless sintering ceramic with highly density and grain size structure [14]. In this work, a small amount of additive was doped into Al_2O_3 compacted pellets as a sintering aid in order to get a desired effect. Basically, an additive such as MgO, TiO_2, ZrO_2, Y_2O_3 and Li_2O_3 are commonly used in ceramic system to influence the densification process either by reducing sintering temperature and time or suppressing and promoting grain growth or enhancing physical and mechanical properties [8,15]. It is well known that a small addition of MgO is favorable to inhibit the discontinuous grain growth and to promote the sintering of Al_2O_3 which leading to completely dense and strongly finer homogeneous structure [1,16]. This finding was also supported with other work by *Soumen Pal et al* showed that MgO enabled the ceramic structure to become more dense [1,10,11,14,16-18]. Futhermore, the MgO additive plays an effective role in sintering of alumina by prevention of the abnormal grain growth in the final stage of densification process which leads to fully densification [15,18]. Therefore in this work, MgO was selected as a sintering aid for the pressureless sintering in consolidating the alumina structure. In the present work also, the effect of MgO with different particle size of compacted powder at different firing and drying condition and cooling rate from the previous studies, to the microstructures and physical properties were investigated and are presented in detail in the next sections.

Experimental

Alumina powder (Al_2O_3-purity of 99%, *Sigma Aldrich*) with particle sizes of 25µm and 90µm were used in the sample preparation of ceramic pellet. The alumina 25µm and 90µm were milled with 0.25wt% magnesia powder (MgO- *ChemAR, Systerm*) separately by ball milling (*Fritch P6, Lab Korea*) for 4 hours, (with using pure Al_2O_3 ball as a milling media).

Then, 1.0wt% binder polyvinyl alcohol, (PVA) was mixed with mixtures of Al_2O_3 and MgO. Pellet samples (approximately 13mm in diameter) were produced by uniaxially pressing under 8 tone using stainless steel die. The pellets were then dried for 24 hours under room temperature and

presureless sintering were conducted at 1600ºC for 2 hours at heating and cooling rate 2 ºC/min in the programmable furnace. The bulk density and apparent porosity of the sintered specimens were measured by the Archimedes method. The microstructure of the pellet was observed by using *JEOL, JSM-638OLA* scanning electron microscope (SEM).

Density and porosity calculation

$$\text{Density} = \frac{W_d}{W_d - W_s} \times 100\% \quad (1)$$

$$\text{Porosity} = \frac{W_w - W_d}{W_w - W_s} \times 100\% \quad (2)$$

Results and discussion

Bulk density and apparent porosity. Table 1 contains a summary of results comparing the bulk density and apparent porosity values for the sintered Al_2O_3 pellets with and without MgO additive. The results show that the pellet sintered for 2 hours with particle size 25µm has a higher bulk density value compared to the pellet with particle size 90µm. This was agreed with the previous observation [4,9] which showed that the smaller particles size of ceramic powder will increase the densification of the ceramic structure. Data in Table 1 also shows the changes of the apparent porosity with different particle size of alumina pellets. Again, the use of the 25µm particle results in the appearance of lower apparent porosity value.

The densification process not just effect by the sintering process but also effect of the solid solution additive. In fact, the value of higher density is difficult to achieve in solid state sintering due to the coarsening process dominate in the densification process. It was proved by the previous study; the use of additive has a significant influence on the density of the microstructure [1].The same phenomenon is also shown with the addition of MgO for both different alumina particles. It was noticed that alumina doped MgO is efficient in reducing the apparent porosity and enhancing the bulk density of the compacted Al_2O_3 structure. These results indicate that the MgO can accelerate and promotes the densification during sintering stage and finally lead to the reduction porosity value of the consolidated structure.

Table 1: Bulk density and apparent porosity for the sintered Al_2O_3 undoped and MgO doped samples**.**

Particle Size(µm) / Sample	25		90	
	Porosity (%)	**Density (g/cm^3)**	**Porosity (%)**	**Density (g/cm^3)**
Al_2O_3	19.9511	3.5588	19.3530	3.5249
Al_2O_3 + MgO	10.1793	3.6777	19.1961	3.6049

Effect of MgO on Microstructure Development. The differences in microstructure changes for each sample after sintered at 1600^{o}C in a closed air system for undoped and doped alumina are shown in Fig.3 and Fig.4. As can be seen in both these figures the addition of MgO can promote the densification and normal grain growth for alumina and results in the stabilization of the microstructure and homogenous densification. As presented proved by previous works [1] showed that MgO can stabilize the microstructure with homogenous densification. It is due to the its ability to suppress grain growth distribution by allowing the porous region to densify without occurrence

abnormal grain growth with the same manner as presented in the previous studies by *M.P.Harmer et al* [18]. These can be clearly seen in Fig.3 and Fig.4 where the addition of 0.25% MgO for different particle size of alumina gives a positive result with a uniform grain structure and homogenous microstructure. These surface sintered samples also revealed that the microstructure of MgO doped alumina show a good bonding structure with a smooth stacking particles indicates a good diffusion process, as compared to alumina undoped MgO showing an obvious separation of pore grain boundary with abnormal grain growth. These situations strongly influences the increment of densification values and the declination of porosity values in the alumina microstructure (as presented in Table 1) as the additive is added for both particle sizes.

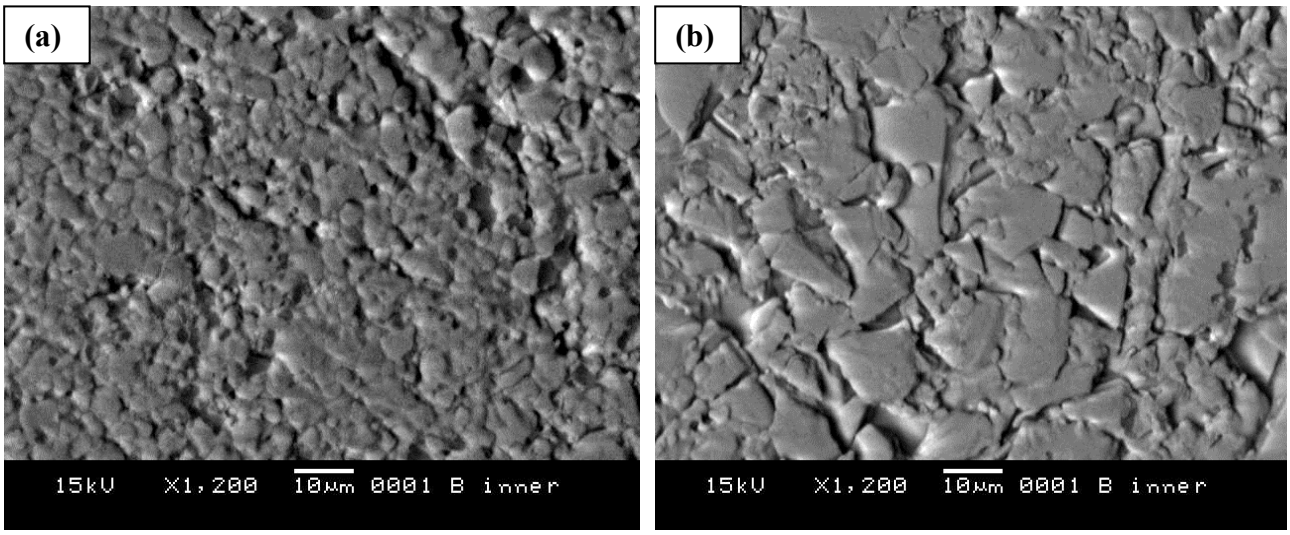

Fig.3. Microstructure development of (a) 0.25% MgO undoped alumina and (b) 0.25% MgO doped alumina with particle size 25µm

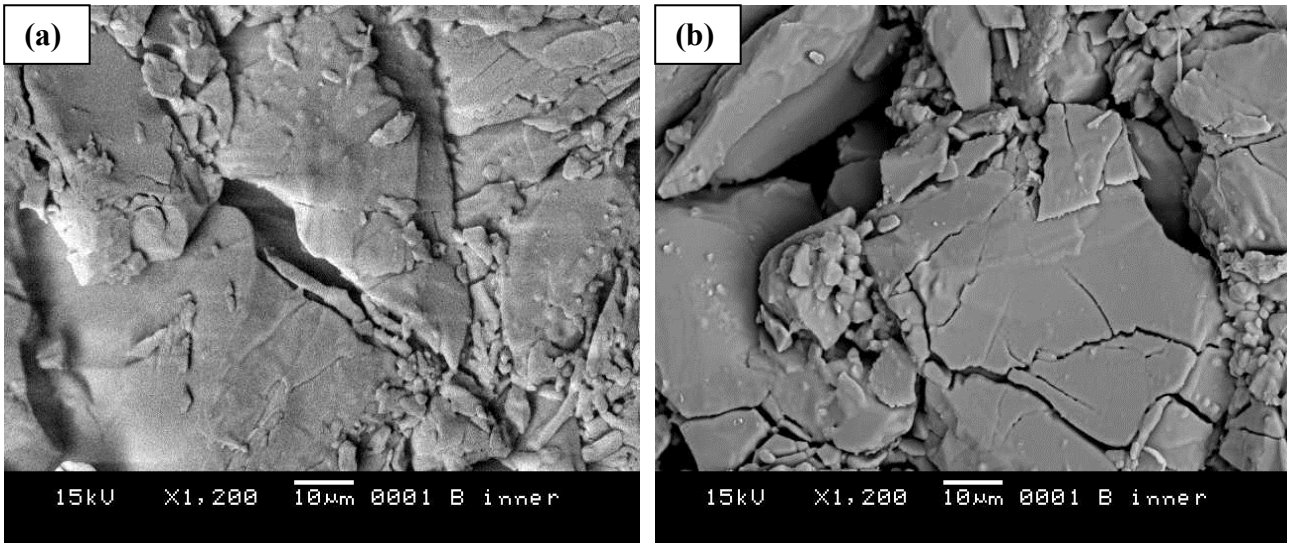

Fig.4. Microstructure development of (a) 0.25% MgO undoped alumina and (b) 0.25% MgO doped alumina with particle size 90µm

Conclusion

In this work, the bulk density, apparent porosity measurement and microstructure observations were used to evaluate the influence of the additive on the sintering properties for the alumina structure with different particle sizes. Based on the observation and measurement that has been carried out it showed that the smaller particles size lead to the dense and lower porosity value of the final structure

formation. In fact both the densification and reduction of porosity can be accelerated with the addition of small amount of sintering aid, MgO, and this is clearly shown in both particles size as presented in Table and Figures above.

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