Effects of Double Sintering on The Properties of Porous Ceramic

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ABSTRACT

This present work reports the fabrication of porous ceramic through thermal treatment of powder mixture of alumina and starch. The effects of double sintering on the properties of porous ceramic were investigated. The mixture was milled and then the dried powder below 63 μm was sintered in powder form at 1100°C, 1200°C, 1300°C, 1400°C and 1500°C. Meanwhile, the second stage sintering at 1500°C was implemented after powder pressing. All sintering process was carried out for 2 hours. The mechanical and physical properties such as porosity, density, modulus of rupture and hardness have been analyzed. The modulus of rupture has risen up to 14.704MPa which has successfully improved it about five times higher compared to the strength for single sintered sample. Besides that, the hardness value for double sintered porous ceramic could be increased until 35.90HV. The results have proven that double sintering process able to improve the density, hardness and strength of porous ceramic significantly. These ceramic bodies may have a potential use in a wide range of applications due to an excellent combination of properties.

Key Words: sintering, porous ceramic, density, hardness, strength

1.0 INTRODUCTION

Almost all ceramic materials have been made through the powder route, and also a variety of components used in modern technology has undergone sintering as one of the production steps. Sintering is one of the most important processes for the fabrication of ceramic materials [1]. The properties of ceramic materials can be modified through sintering process. The complexity of the sintering process makes this field a fascinating area for research.

The process by which small powder particles of a material are bonded together by solid-state diffusion is called sintering. In ceramic manufacturing, this thermal treatment results in the transformation of a porous compact into a dense, coherent product. In sintering process, particles are coalesced by solid-state diffusion at very high temperatures but below the melting point of the compound being sintered [2,3]. During the sintering of a powder compact, both densification and grain growth occur simultaneously [4]. In order to understand and control the sintering process, the relationship between densification and grain growth must be assessed. It concerns the materials constitutive laws governing the sintering process which is related to the microstructural evolution of the sintering component [5].

Usually, the preparation of ceramic body involves only single sintering process. The sintering process for ceramic preparation involves powder compaction method will be done after the pressing and drying process. However, in the present work, alumina ceramics which is prepared by powder route has gone through sintering process twice in order to produce porous alumina ceramic body.

2.0 EXPERIMENTAL

Alumina and starch powder with particle size below 63 μm were used in this study. The mixture of raw materials based on volume percentage was milled for about 4 hours. Distilled water was used for preparation of slurries. The ground slurry was dried in an oven and sieved on a 63 μm screen. The undersize powder was then sintered in a temperature of 1100°C, 1200°C, 1300°C, 1400°C and 1500°C in the form of powder. The sintered ceramic powder was form into a green body by uniaxial pressing at 10 tonne to form rectangular bars. The purpose of polyvinyl alcohol addition during pressing was to ease the sample fabrication process. The rectangular bars were re-sintered in the air furnace at 1500°C for 2 hours with a ramp of 2°C/min. Figure 1 shows the controlled temperature profile of the furnace during heating and cooling of the raw mixture and the pressed bar samples. Meanwhile, the sequence of operation for preparing porous ceramic is depicted in Figure 2. A sample without re-sintering which undergone only single sintering temperature at 1500°C was also prepared for comparison of data with re-sintering samples.

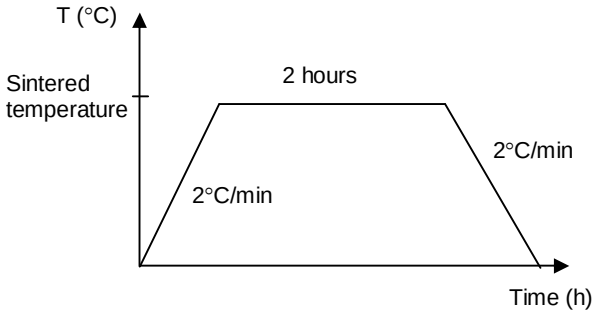

Figure 1: Sintering profile of porous ceramic

The sintered porous ceramic bodies were characterized by their physical and mechanical properties such as apparent porosity, density, flexural strength and hardness. Three-point bend strength was measured using Autograph AG-I Universal Testing Machine with applied load at a cross head speed of 1.0 mm/min. As for hardness test, the microhardness machine

(HMV-2000 Shimadzu) was used. Apparent porosity and bulk density were measured by water immersion technique according to Archimedes' principal.

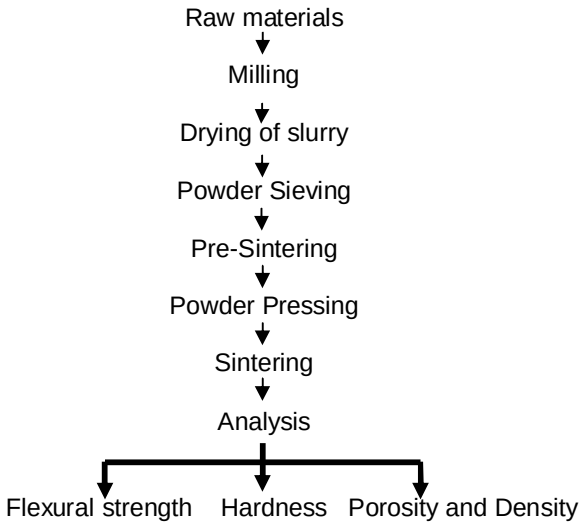

Figure 2: Flow chart for the sample preparation and testing of porous alumina ceramic

3.0 RESULTS AND DISCUSSION

3.1 POROSITY AND DENSITY ANALYSIS

Porosity is the dominant factor affecting the performance of ceramic materials. From the past research, the use of different starting alumina compounds, the choice of sintering temperature and hold time, as well as heating rate, and the introduction of sintering aid provide the opportunity to control the formation of pores size and porosity distribution in the wide range. The shrinkage during the sintering process tends to the simultaneous decrease of the porosity of the sintered samples [6].

Table 1 shows that porosity decreases with the increment of powder sintering temperature. The porosity drops gradually from 31.15% to 25.93%. This phenomenon is due to elimination of pores between aggregates and agglomerates and a fast grain growth during sintering. Cooled at lower temperature with longer dwelling time will make the porous ceramic body denser. Besides that, ceramic materials would like to get rid of all its grain boundaries so that it would have the lowest possible energy state.

Table 1: Porosity and density of porous alumina ceramics

	Powder sintering temperature °C (Samples from pre-sintering process)					Samples without pre-sintering
	1100	1200	1300	1400	1500	
Apparent Porosity (%)	31.15	29.01	28.03	27.55	25.93	39.29
Bulk Density (g/cm^3)	1.173	1.226	1.248	1.261	1.278	1.226

The result indicates that the cell size in ceramic foams is very sensitive to the baking condition and accelerates the cell coarsening [6]. Therefore, the controlling mechanism for porosity can be the alumina grain growth during firing.

In stark contrast to the characteristic of porosity, the bulk density of alumina foam increases with the rising of powder sintering temperature. The steadily rose of bulk density is caused by the declination of porosity in ceramic body. Samples without pre-sintering have a bulk density of 1.226 g/cm^3. However, the same value of density is obtained from the ceramic powder which has gone through pre-sintering at 1200°C. At higher pre-sintering temperature of ceramic powder, the increment of bulk density can be achieved.

3.2 FLEXURAL STRENGTH

Figure 3 shows the graph of flexural strength versus alumina loading. Flexural strength for samples which undergo pre-sintering process significantly exhibits higher value compared to samples without pre-sintering. Alumina porous that has gone through pre-sintering at 1500°C gave the highest strength 14.704 MPa which is almost five times higher than alumina porous without pre-sintering with the strength of 3.096 MPa. The implementation of pre-sintering process obviously contributes to drastic improvement of the strength of alumina porous body.

The changes in flexural strength can be related with the decreasing of porosity in sintered alumina body. Porosity has a significant role to influence the flexural strength of sintered alumina porous body. It is well known that the strength of the porous ceramics increases with the decreasing of porosity [7]. Figure 4 shows the effect of porosity on flexural strength for the sintered alumina foam.

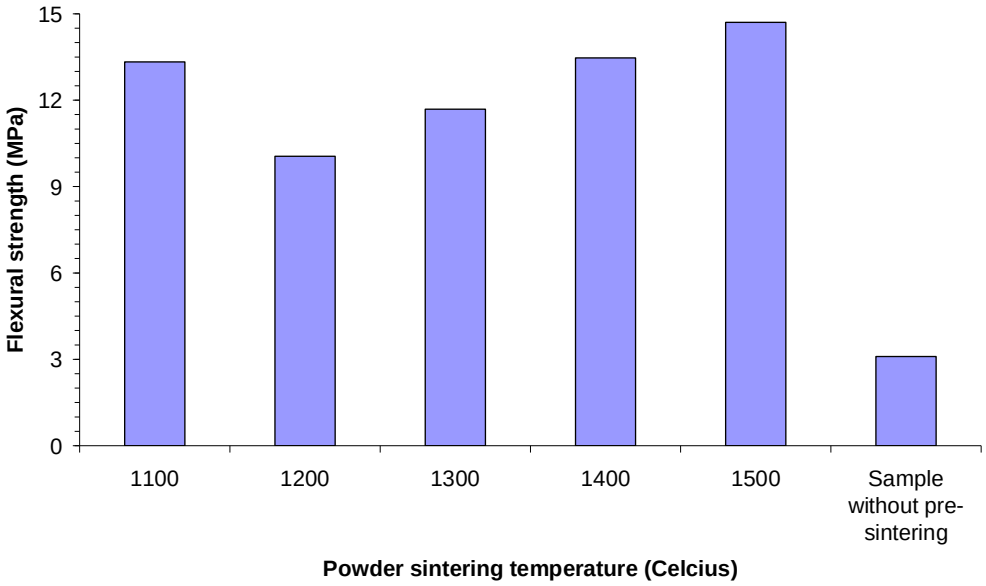

Figure 3: Flexural strength of alumina porous ceramic at different powder sintering temperature and sample without pre-sintering

The flexural strength is inversely proportional to the porosity [8,9]. Previous works indicated that changes of porous body strength as a result of grains grew up that led to the porous body had more contacting areas. It became the main reason for causing the change of foams strength at that time.

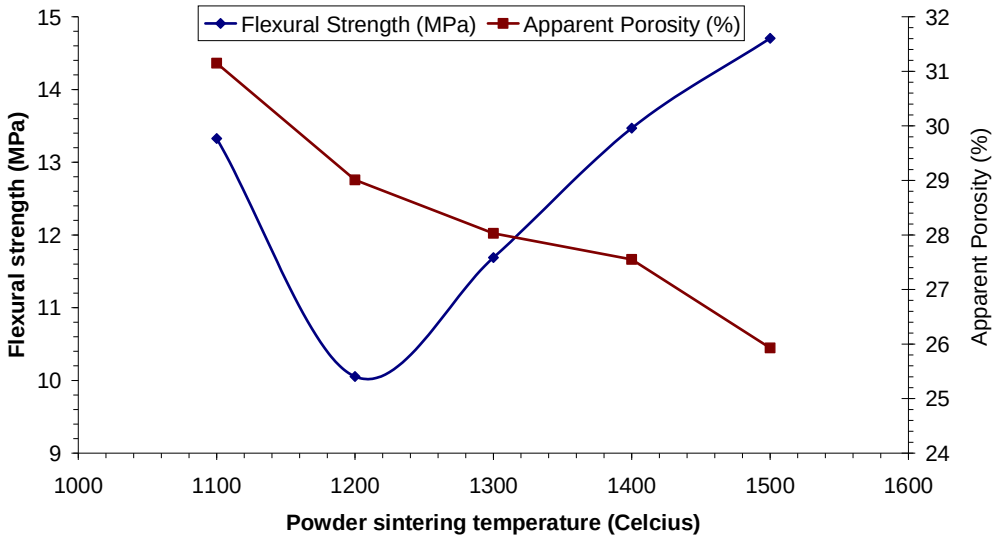

Figure 4: Correlation between flexural strength and porosity of sintered ceramic foams.

3.3 HARDNESS

Results in Figure 5 depicts there is a wide gap between hardness value of sample with and without pre-sintering process. Sample without pre-sintering only gives 2.5 HV. Meanwhile, the hardness value for pre-sintering alumina porous body has risen rapidly. This situation may be due to the powder refinement as the pre-sintering temperature becomes higher. The decrement in porosity of porous body also contributes to the improvement of mechanical properties such as hardness.

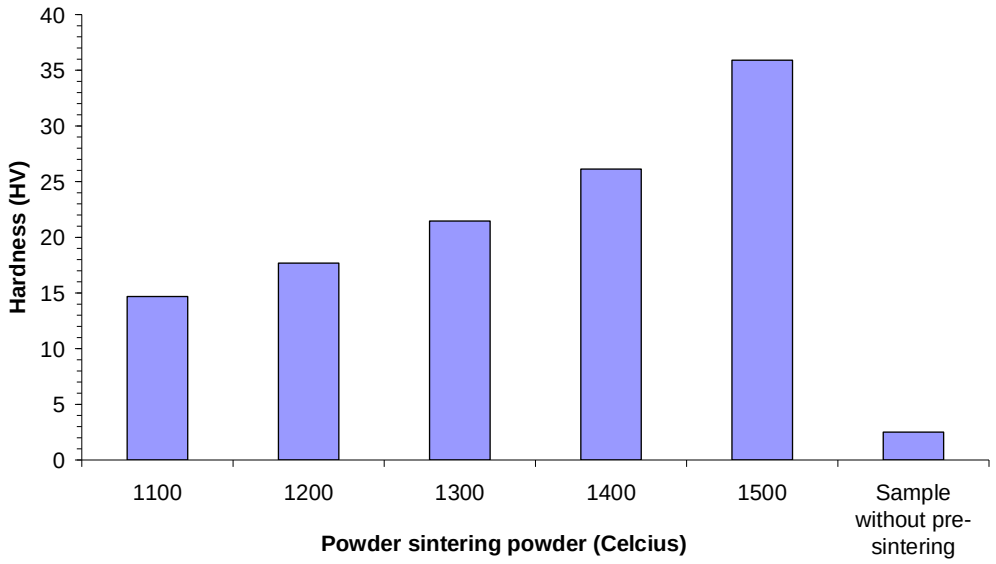

Figure 5: Hardness of alumina porous ceramic at different powder sintering temperature and sample without pre-sintering

Pre-sintered sample at 1100°C has successfully improved the hardness value to 14.68 HV which is about six times higher than the hardness of sample without pre-sintering. Powder sintering temperature from 1100°C to 1500°C managed to rise up the hardness in the range of 14.68 HV to 35.90 HV.

4.0 CONCLUSION

Powder sintering of alumina by the addition of starch has proven to enhance the density, strength and hardness of alumina porous body significantly. Pre-sintering at only 1100°C enable the production of alumina porous body by press forming which gives 31.15% porosity with sufficiently higher hardness 14.68 HV and strength of 13.33 MPa. Such ceramic bodies may have a potential use in a wide range of applications due to an excellent combination of properties. The success can be explained by the pre-sintering of mixtures in the powder form, as the sample without pre-sintering produced much lower porous body characteristics.

5.0 ACKNOWLEDGEMENT

The authors would like to thank the financial support from Universiti Tun Hussein Onn Malaysia through vot 0152 for the Fundamental Research Grant.

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