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The influence of sintering temperature on characteristic of ceramics based on bentonite, glass bead and alumina

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Abstract. Bentonite, glass bead, and Al_2O_3 based fine ceramics with variation of sinter temperatures (900, 1000, 1100, and 1200°C) have been prepared by powder metallurgy method. The preparation process of raw materials starts from the mixing of 35 wt% powder bentonite, 35 wt% glass bead, and 30 wt% alumina using High Energy Milling (HEM). After that, the powders are compacted with 80 kgf/cm^2 into pellets. The samples are dried using oven at 100°C for 24 hours. Characterization tested includes physical properties (density, porosity, water absorption, and hardness), microstructure by using SEM and phase analysis by using XRD (X-Ray Diffraction). The results show that the phase formed are the major phase of albite ($Al_2NaO_8Si_3$) and quartz (SiO_2), while the corundum (Al_2O_3) and nepheline ($AlNaO_4Si$) are minor phases. The result of analysis shows that bulk density and hardness value tends to increase, while porosity and water absorption tend to decrease along with increasing sinter temperature. The optimum condition was reached at 1100°C with bulk density = 2.43 g/cm^3, porosity = 1.91%, water absorption = 0.8, and hardness = 878.29 HV.

1. Introduction

Ceramic materials based on oxide compounds like Al_2O_3, ZrO_2, MgO, and TiO_2 have advantages such as high melting point, hard, refractory (high temperature resistant), strong, and insulators [1, 2]. Aluminum oxide (alumina) is a chemical compound of aluminum and oxygen, with the chemical formula of Al_2O_3. This material melts at a temperature of 2050°C and maintains its strength even at a temperature of 1500 to 1700°C [3, 4]. Alumina is often used in various applications because of its strict physical and chemical properties such as very high strength, extremely hard, good electrical insulation, high heat resistance, high overtime temperature, high abrasion, and corrosion resistance [5, 6].

Silica or silicon oxidation has a chemical formula SiO_2 formed from silica and oxygen atoms. Silica can be applied in various mechanical electronics and can be made into ceramic materials. Silica compounds contained in nature are crystalline structures, which as compounds derived from synthetics are amorphous. Silica can be found in three crystalline forms that is quartz, tridymite, and cristobalite [7, 8].

The sintering process is a process of compacting powder material by forming grain boundaries bonding between powder constituents. The intercellular bonding occurs due to heating and sintering temperature set under the melting temperature of the constituent particles. In the sintering process, compacted materials occur because of the formation of bonds between particles [9].

In this experiment, the research will be focused on influence of sintering temperature variations on the characteristics of ceramics based on alumina, bentonite, and glass bead. Samples in the pellet form are sintered at 900, 1000, and 1100°C [10-12]. Characterization is performed to find out crystallinity, density, hardness, and microstructures.

2. Experimental Methods

The preparation process began with the preparation of Al_2O_3 (30 wt%), glass bead (35 wt%), and bentonite (35 wt%) materials [13]. All the materials were mixed and milled with an aquadest medium by using HEM (High Energy Milling) for 2 hours. The milled powder was then heated to 100°C for 24 hours. The fine powder was put into beaker glass and was added 5 wt% of epoxy resin as adhesive. The mixture was sintered at the temperature of 900, 1000, and 1100°C for 4 hours, which were named as samples A, B and C, respectively. The characterization includes particle size distribution by using particle size analyzer (PSA Cilas-1190), phase analysis with X-ray diffraction (XRD Rigaku Smart Lab type, λ = 1.5418 Å), microstructural analysis with scanning electron microscopy (SEM Hitachi SU -3500), and hardness analysis with microhardness tester (MHT LECO LM-100AT)

3. Results and Discussion

The powder particles size distribution of bentonite, glass beads, alumina, and mixed powder material are shown in figure 1. The original particle size of bentonite, glass beads, and alumina is 16.88, 516.97, and 105.86 µm. After milling for 2 hours, the average particle size is 4.27 µm, as shown figure 1. From figure 1, it shows that the particle size is less homogeneous distribution. For 10% cumulative distribution, there is a particle diameter about 0.2 µm which are part of the bentonite. While particle diameter between 0.8 - 20 µm is the largest part of alumina and glass beads. From previous study, for a similar composite has similar range of silica and alumina diameter is around 6–50 µm [14].

The bulk density and porosity of samples A, B, and C which was sintered at the temperature of 900, 1000, and 1100 ° C for 4 hours respectively were measured by using Archimedes method (ASTM C373-88) [15, 16]. From Figure 2, it can be seen that the density value is inversely proportional to the porosity. The higher of sintering temperature increases the density and decreases the porosity of the samples. As a result of sintering, densification process was occurred and resulted in less number of pores [17]. The highest density and porosity value are on sample C, with the value about 2.43 g/cm^3 and 1.92%, respectively because the phases of corundum and quartz are occurred as can be seen in figure 3.

The result of XRD analysis is shown in figure 3. From figure 3, it shows that in samples A and B have identical phase. There are 4 phases of albite ($Al_2NaO_8Si_3$), corundum (Al_2O_3), nepheline ($AlNaO_4Si$), and quartz (SiO_2) [18]. While on sample C, it shows that the peak of nepheline and quartz are reduced while albite's peak is sharper. This suggests that the effects of quartz and nepheline change to form the albite phase.

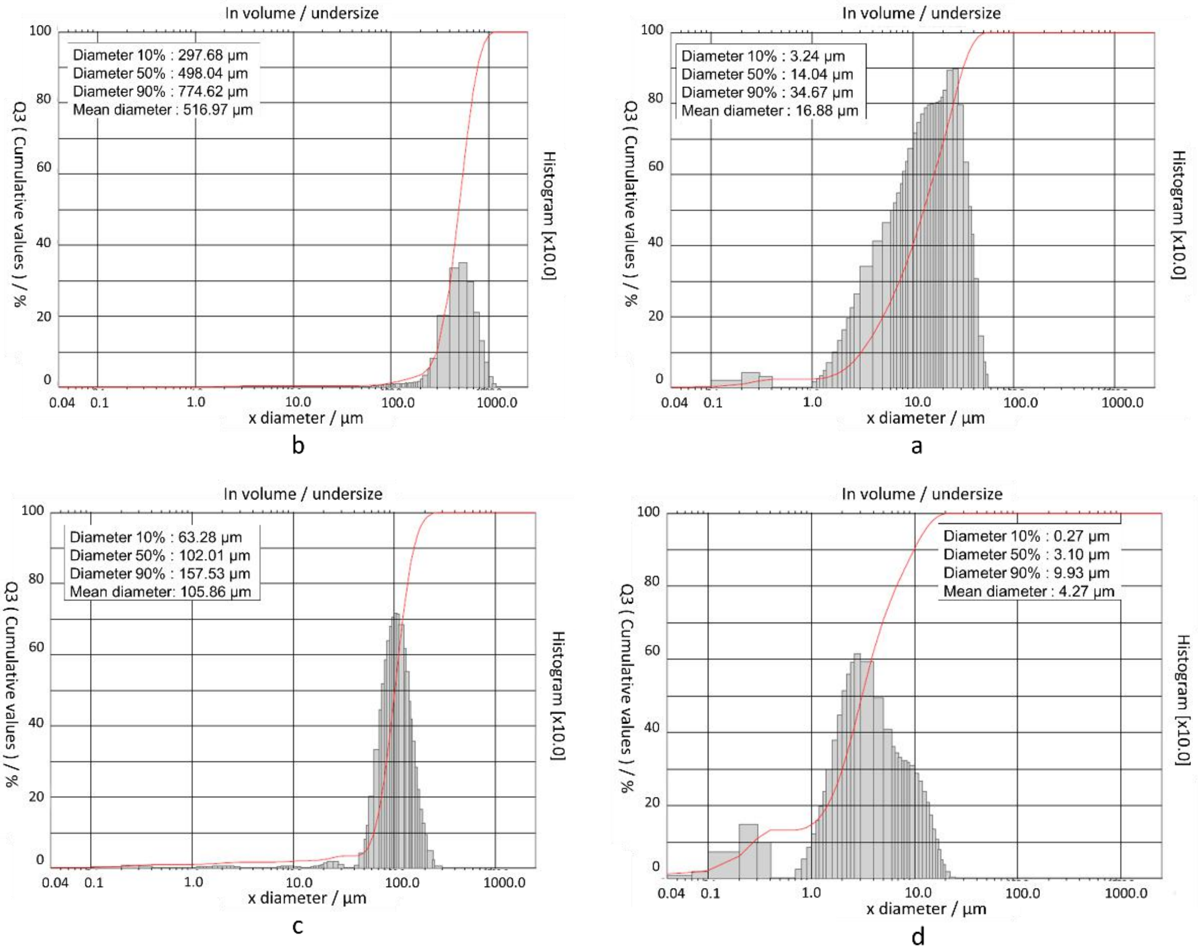

Figure 1. Particle size distribution of (a) bentonite, (b) glass beads, (c) alumina and (d) mixed sample after milling for 2 hours.

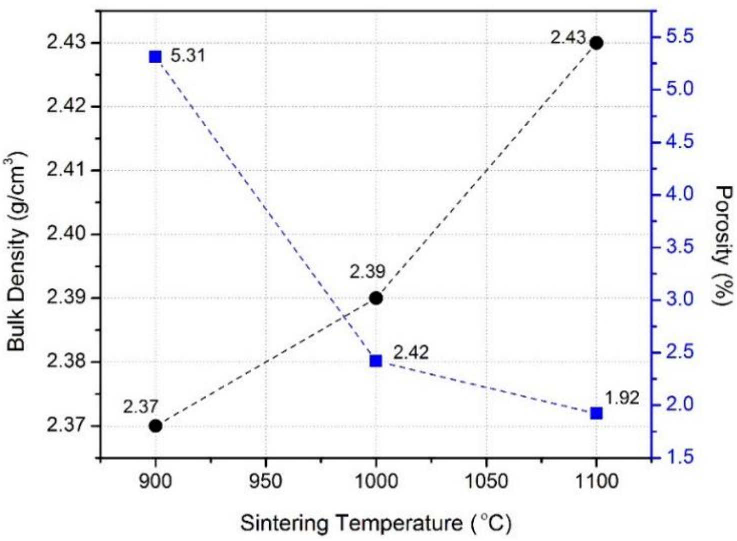

Figure 2. The influence of various sintering temperature to bulk density and porosity of ceramic based on bentonite, glass bead, and alumina.

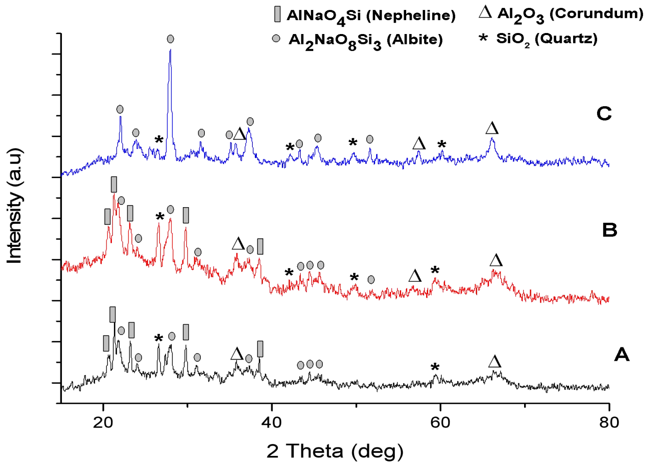

Figure 3. X-ray diffraction pattern of ceramic based on bentonite, glass bead, and alumina with various sintering temperatures.

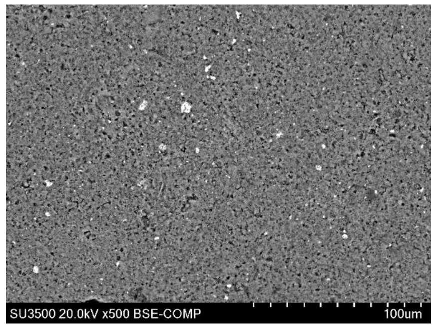

Figure 4. SEM images of ceramic based on bentonite, glass bead, and alumina after calcination of 900ºC (sample A).

Sample morphological analysis with SEM is shown in figure 4. In this research, the morphology of sample A is analyzed by SEM. From figure 4, it shows that in higher of sintering temperature, pore of the sample is getting smaller. This is consistent with the results of its density and porosity measurements. The higher of sintering temperature causes the higher of density but the porosity tends to shrink [14]. From the result of SEM-EDX, there are elements of O, Na, Mg, Al, Si and Ca in the sample A. The SEM-EDX results related to XRD results with albite ($Al_2NaO_8Si_3$), corundum (Al_2O_3), nepheline ($AlNaO_4Si$), and quartz (SiO_2) phases. Even though, there does not find an element of Ca in the phase from XRD results. This is due to Ca as a minor content in the amorphous form which Ca is part of bentonite [11, 15].

The hardness test to finding the influence of sintering temperature on ceramics based on bentonite, alumina and glass bead is measured by using Vickers method. The hardness test results are shown in figure 5. From figure 5, it can be seen that the value of hardness increases with the increase of sintering temperature. In this study, the highest hardness was obtained in sample C with the hardness value of 878.29 HV. The hardness degree of material is strongly influenced by the density and porosity. The higher the density of a sample increases the hardness of the sample. The result of the hardness in these samples is corresponding to density and porosity of the samples. Sample C that has higher density and lower porosity has higher hardness and vice versa. Meanwhile, as the result of diffraction pattern, is albeit which more hardness than nepheline and the dominant phase in component of sample C, so it caused sample C to be the hardest among others.

4. Conclusions

It has been made fine ceramic based on alumina, bentonite, and glass beads with a sintering temperature of 900, 1000 and 1100°C for 4 hours. The characterization results show the sample density increases, while the porosity decreases with the increase of sintering temperature. The result of phase analysis shows that there are 4 main phases: albite ($Al_2NaO_8Si_3$), corundum (Al_2O_3), nepheline ($AlNaO_4Si$), and quartz (SiO_2). The value of sample hardness increases with the increase of sintering temperature. The optimum sample was obtained at the sintering temperature of 1100°C (sample C) with density, porosity, and hardness about 2.43 g/cm^3, 1.92%, and 878.29 HV. Therefore, the best composition for fabrication of fine ceramic based on alumina, bentonite, and glass beads is the sample C.

Table 1. Element analysis result of ceramic based on bentonite, glass bead, and alumina after calcination at 900°C (sample A).

Element	Weight%	Atomic%
O	64.64	76.05
Na	2.28	1.87
Mg	1.38	1.07
Al	13.89	9.69
Si	14.81	9.92
Ca	3.00	1.41
Totals	100.00	100.00

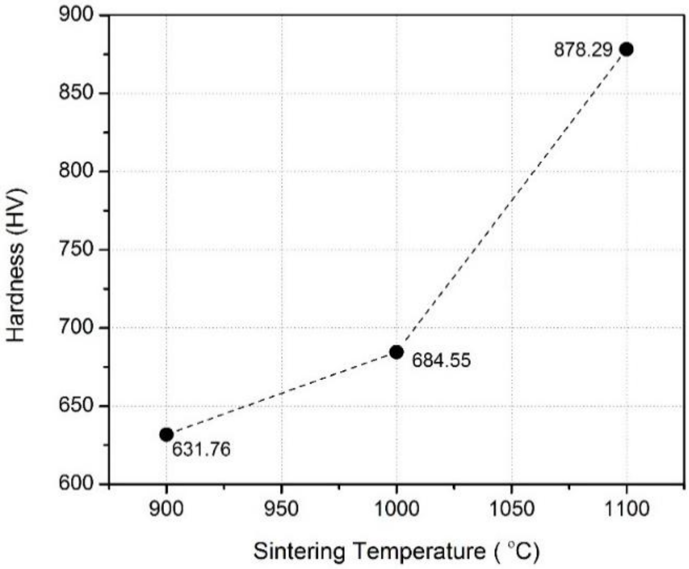

Figure 5. The influence of various sintering temperature to hardness of ceramic based on bentonite, glass bead, and alumina.

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