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Characterization of Alumina Foam Prepared by Space Holder Method

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Abstract: Ceramic foams can be categorized as brittle and porous and composed of many pores inside compared to other metals and polymers. Due to their unique properties, ceramic foams have been widely used in various applications such as thermal insulation, filtration and biomedical implants. In this research, the ceramic foam which is alumina foam was fabricated through the space holder method. The raw materials used were alumina powders, Carboxymethyl cellulose (CMC) and Polyethylene glycol (PEG) as binders and coconut husk (CH) powder as space holder materials. The coconut husk powder varied in the different compositions of 0, 5, 10, 15, 20 and 25 wt %. CMC and PEG binders act as a glue that attaches all the raw materials resulting in a strength increase of alumina green samples during compaction. All the raw materials were then mixed, ball-milled and compacted into a solid cylinder sample of 0.7 cm in height. Next, the alumina green samples were sintered at a temperature of 1350 ˚C in an electrical box furnace. Several types of tests were also conducted to analyse the physical and mechanical properties of alumina foams that were fabricated with different coconut husk powder compositions. Microstructure observation using Scanning Electron Microscope (SEM) was conducted that shows the alumina foams have single and interconnected open pores with pore sizes in the range of 2 to 100 μm. From the results, it was found that the density decreased, and the porosity increased in the range of from 1.7936 to 1.4781 g/cm^3 and 36.16 to 57.66% respectively. As the density decreased, the compressive strengths were found to decrease from 1.45853 to 0.35174 N/mm^2. In comparison to others, the lowest CH powder was the ideal space holder composition to be use as alumina foam because it has the smallest shrinkage, largest density, smallest porosity percentage and highest compressive strength.

Keywords: Alumina Foams, Coconut Husk Powder, Space Holder Technique

1. Introduction

Ceramic foams or cellular ceramics are tough foam or foam-like structure that was originally made from ceramics with unique properties in combination such as chemical and thermal stability, high melting temperature, high corrosion resistance and biocompatibility. In addition, the presence of the pores results in lower density, increased surface area, high fluid permeability, reduced thermal

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conductivity and increased specific strength[1], [2]. Due to their attractive properties, ceramics foams are widely used in many industries such as filtration of molten or hot gasses, thermal protection systems, catalyst supports, absorbents, chemical sensors, biomaterials and gas combustion burners [3], [4].

This project aims to study the potential use of coconut husk powder as s space holder material in the fabrication of porous ceramic and to investigate the physical and mechanical properties of alumina foam where the shrinkage analysis, density and porosity, microstructure analysis and compression test will be done. The possible outcomes from this project will be a simple and cost-effective method in alumina foam fabrication that will be beneficial for industrial usage. The procedure of this method can be divided into three main parts, which are the mixing of space holder material, binders and alumina powder, compaction of the mixed substances, and sintering process at a high temperature below the alumina foam melting point.

The use of coconut husk powder as a space holder particle in the alumina foam fabrication using space holder method and the characterization of alumina foam will be investigated in this research. The significance of this project is that this project could be a benchmark for a cost-effective and simple method in ceramic foam fabrication for the industry.

2. Research Methodology

A full explanation about the experimental procedure used throughout the course of the research. The process consists of material selection, sample preparation, characterization techniques and equipment used for analysis. All the apparatus and procedure for this research also written in this chapter according to the methodology flowchart provided in Figure 1.

2.1. Materials and sample preparation

A commercial alumina powder, Al2O3 powder (ρ= 3.95 g/cm^3) with high purity 99.8% was used as starting material, 5 wt% of each Carboxylmethyl Cellulose (CMC) and Polyethylene Glycol (PEG) powders will be used as binders and the CH powder purchased from domestic market, ground using ordinary electrical grinder and sieved to a particle size ≤200 ηm was added with ratio of 0, 5, 10, 15, 20 and 25 wt% and ball-milled for 30 mins on ceramic jar. Cylindrical (diameter= 13 mm, height =7 mm) for characterization were produced using uniaxial compression at load of 9 tones for 2 minutes unsing manual hydraulic press (Carver, maximum load of 11 tons). For each CH compositions, 6 samples were prepared. All the samples were sintered in a box furnace at temperature of 1350 ˚C with 2 ˚C/min and 5 ˚C/min heating and cooling rate respectively.

2.2. Data Analysis

Microstructure analysis: Pore distribution and size of Al_2O_3 foam was examined using JSM 6380LA (JEOL, Malaysia) scanning electron microscope (SEM). *Shrinkage analysis:* The linear shrinkage of Al_2O_3 samples before and after sintering was calculated using formula below:

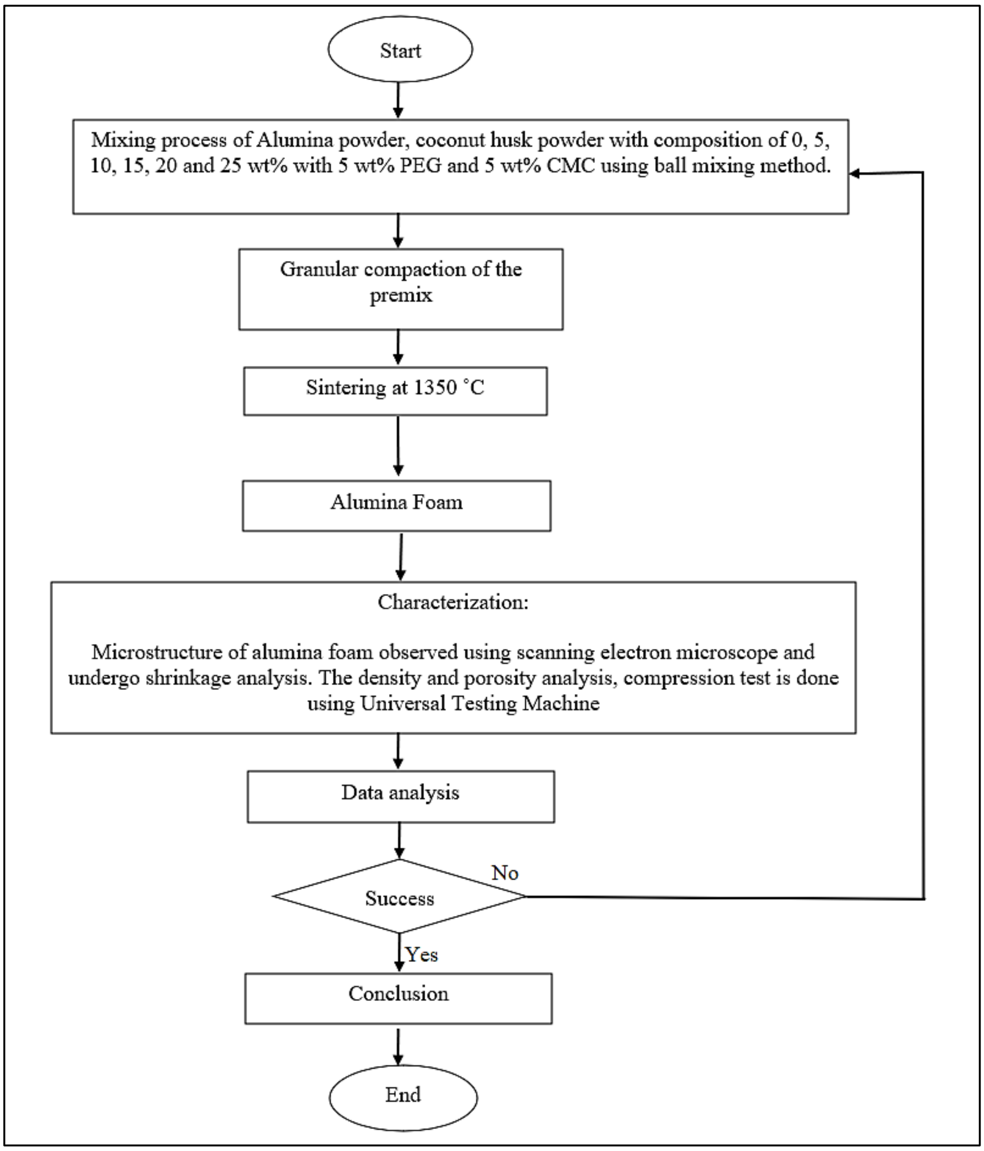

Figure 1: Methodology flow chart

$$\Delta x = \left|\frac{x_1 - x_2}{x_1}\right| \times 100\%$$

Where x_1 and x_2 is height measured before and after sintering. *Porosity and density analysis:* Porosity of Al_2O_3 foam was determined according to standard ASTM C 271-94 whereas Archimedes principle was applied in porosity analyzation [5] using Mettler Toledo X64 and formula below were used.

$$\text{Density, } \rho = \frac{Wd}{Ww - Ws}$$

$$\text{Porosity, } \eta = \frac{Ww - Wd}{Ww - Ws}$$

Where W_d is dry weight of the alumina foam, W_w is wet weight of the alumina foam after immersed in the water and W_s is weight of the alumina foam when suspended in the water. *Mechanical testing:* The compressive strength of Al_2O_3 foam were determined using Universal Testing Machine (UTM) according to ASTM C773-88 standard at a speed 5mm/min.

3. Results and Discussion

The characterization testing of alumina foam fabricated by different composition of coconut husk powder was analyzed by using SEM, density and porosity kit and Universal Testing Machine (UTM) SEM was conducted to study the pore distribution and pore size of the alumina foam. Meanwhile, the density and porosity kit and UTM was used to analyzed the shrinkage percentage, densities, porosities and compressive strength of alumina foam, respectively.

3.1 Shrinkage analysis

The shrinkage analysis was done by measuring the mass before and after sintering as the coconut husk powder burnout leaves the pores. As the pores formed in the samples, there is the possibility of the alumina foam sample will shrink, and the alumina foam may be cracked so a very secure precaution needed to be taken. Figure 4.1 shows the graph of shrinkage percentage of each alumina foams.

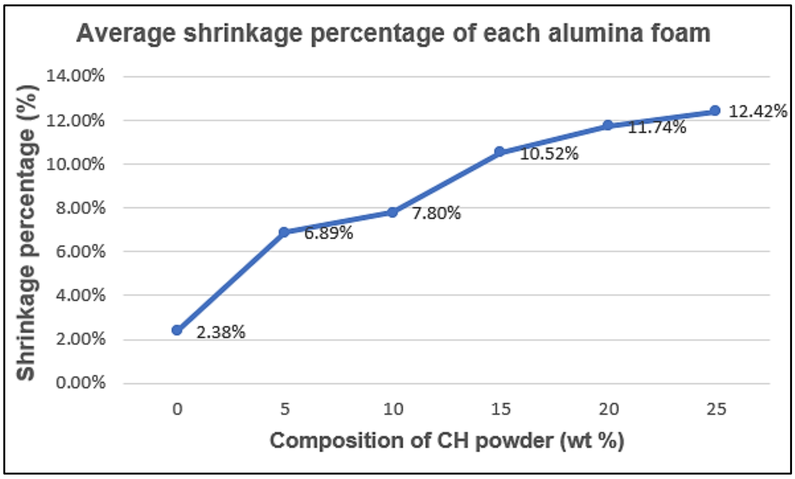

Figure 2: Shrinkage percentage of alumina foams

Figure 2 shows the shrinkage percentage of alumina foam increase proportionally as the composition of coconut husk powder as space holder material increased. The alumina foam that was fabricated with 0 wt % composition of coconut husk powder shows the lowest shrinkage percentage which the height before and after sintering of alumina foam measured. Major difference in increasing shrinkage percentage between 0 and 5 wt % of composition of coconut husk powder can be observed in Figure 4.2 above due to the addition of coconut husk powder.

3.2 Microstructure Analysis

In this part, the microstructure of alumina foam was observed to determine the pore distribution and pore size due to different composition of coconut husk powder used as space holder materials. The magnification of Scanning Electron Microscope (SEM) used was varied to measure and capture the best image of microstructure. The pore size was measure using software ImageJ [6]. Since the alumina foam samples were brittle, the microstructure observation was done only on the top surface of the alumina foam samples as shown in Table 1 below.

Table 1: Microstructure of alumina foam under SEM

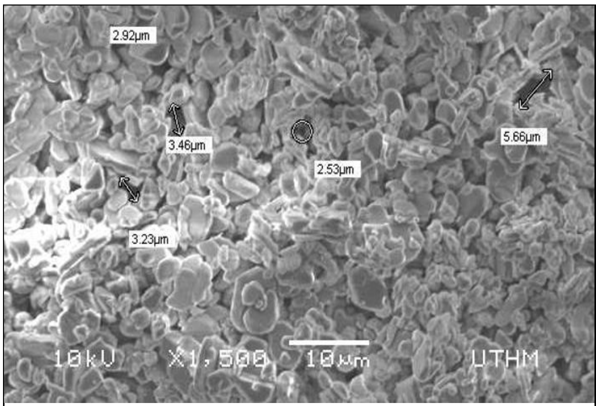

Figure 3(a): Alumina foam with 0 wt% CH powder

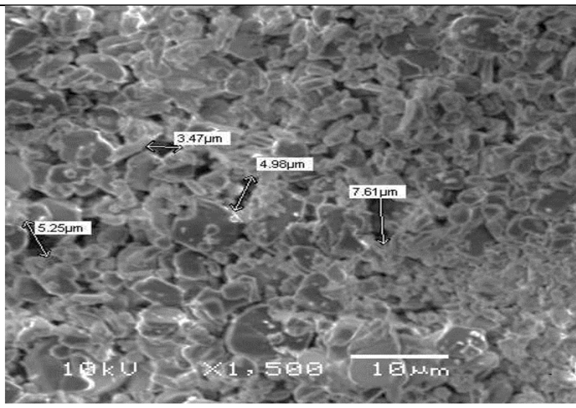

Figure 3(b): Alumina foam with 5 wt% CH powder

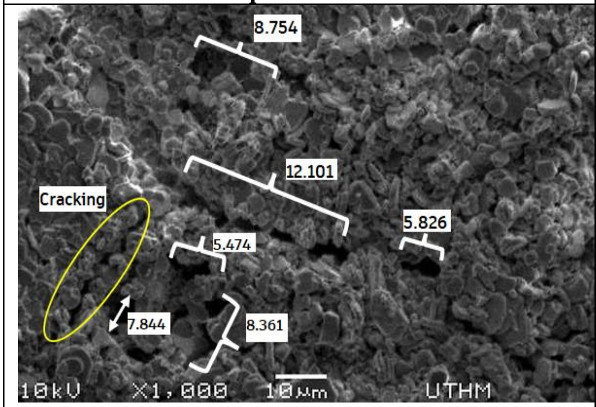

Figure 3(c): Alumina foam with 10 wt% CH powder

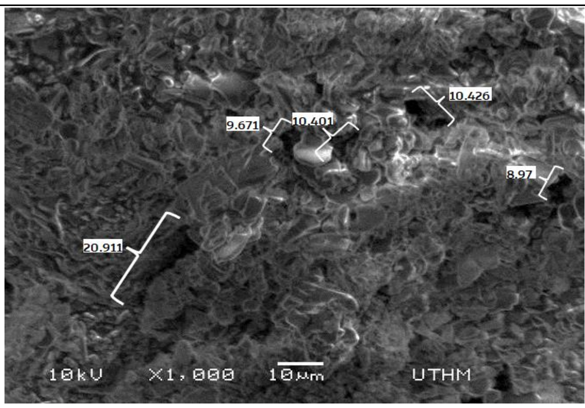

Figure 3(d): Alumina foam with 15 wt% CH powder

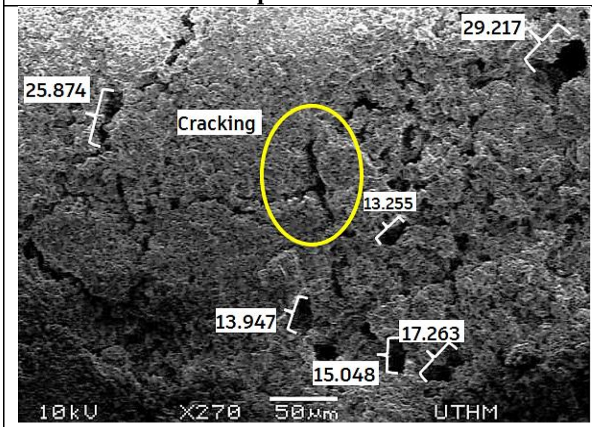

Figure 3(e): Alumina foam with 20 wt% CH powder

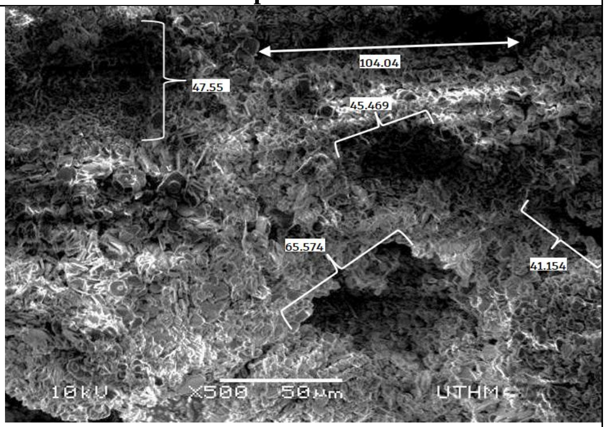

Figure 3(f): Alumina foam with 25 wt% CH powder

Based on Table 1 above, it can be seen that the pores distribution of alumina foam fabricated by different composition of CH powder were not uniformly distributed. The microstructure of alumina foam with no coconut husk powder is more likely to produce single and isolated open pores with a mean pore size of 2.33 µm due to the burnout of binders of PEG and CMC. With the addition of 5 wt% and 10 wt% of CH powder, the increase in single pores and interconnected open pores was observed with the mean pore size of 4.439 µm and 6.845 µm respectively. Cracking can be seen on the surface of alumina foam with 10 and 20 wt% of CH powder may be due to poor control during compaction of green bodies and the sintering process makes the cracks more obvious. As the composition of CH powder increased from 15 to 25 wt%, the mean pore size of open large pores can be observed were 8.5257 µm, 17.381 µm and 46.063 µm respectively. The large pore that existed in the alumina foams may be due to the agglomeration of alumina powders, binders and CH powder during the ball mill process.

3.3 Density and Porosity Analysis

After the green samples were sintered under high pressure, it will leaves pores as a result of burn out of binders and CH powder that led to the samples' shrinkage and decrease in bulk density of alumina foam [7]. This was proved using Archimedes' principle where the dry, wet and suspended weight of alumina foam measured using the density and porosity kit Mettler Toledo XS64. The results of bulk density and porosity of alumina foam fabricated using different composition of CH powder as shown Figure 3 and Figure 4 below.

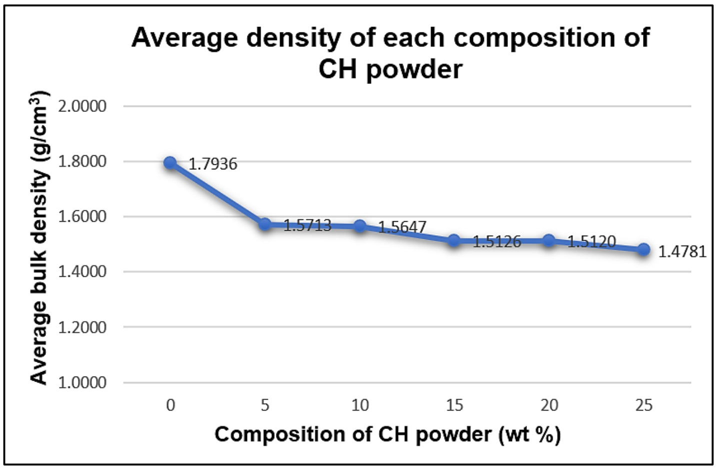

Figure 3: Average density CH powder compositions of alumina foam

The highest bulk density of alumina foam was obviously samples that were fabricated with zero CH powder which was 1.7936 g/cm^3 since the pores that existed in the alumina foam are in single, isolated and small sizes as discussed previously. The density of alumina foam shows a drastic decreasing pattern when the composition of 5 wt% CH powder was added and continued to decrease as the CH powder increased. This also can be proved through previous research that the increase in the composition of space holder materials holds the lower density of ceramic foams [8].

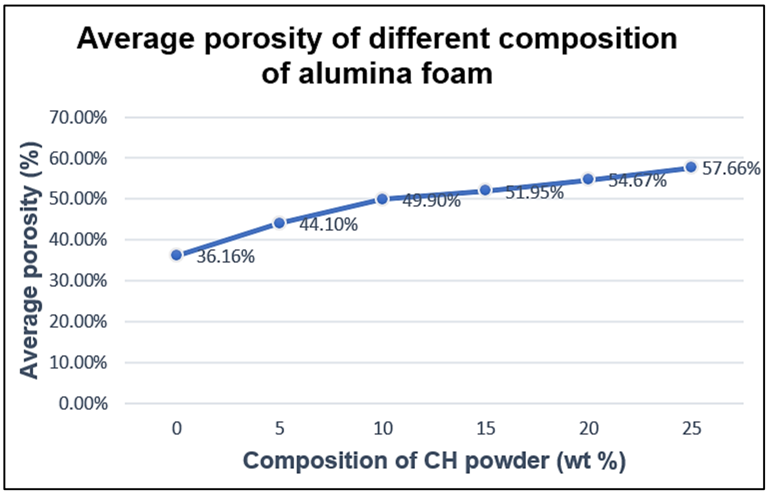

Figure 4: Average porosity of different CH powder compositions of alumina foam

Based on Figure 4, alumina foam with 25 wt% of CH powder has the highest porosity which is 57.66% as it leaves the largest pore size as the binders and CH powder burnt. The agglomeration may contribute to the large pore size so the sieving process to a very fine particle size of CH powder after ball milling is needed. The graph concludes that the porosity of alumina foam increased as the CH powder composition increased.

3.4 Compression Test Analysis

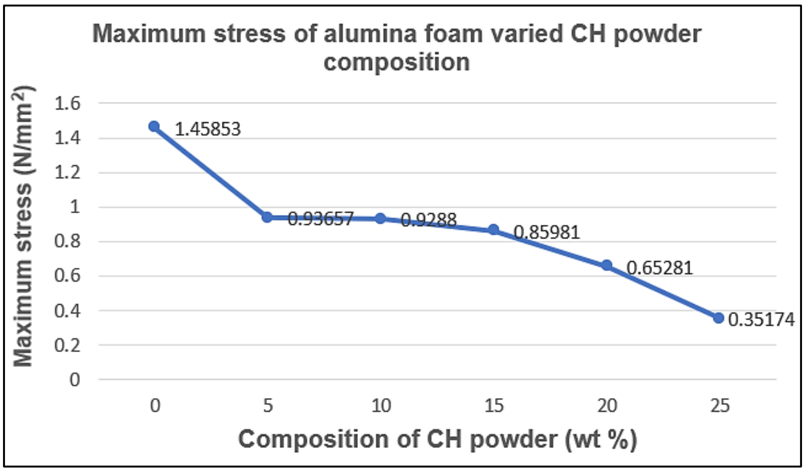

Figure 5: Maximum stress of alumina foam with different CH powder composition

The maximum stress that alumina foam can handle continued to decrease with 10, 15, 20 and 25 wt % of CH powder used as a space holder as illustrated in Figure 5 above. The maximum stress of alumina foam with zero CH powder composition shows the highest value which is 1.45853 N/mm^2 and drops with the addition of 5 wt% of CH powder. This is due to the presence of many voids or pores that cause the shrinkage in alumina foam which concludes the compression strength of alumina foam decreased as the composition of alumina foam increased. Furthermore, the presence of cracks on the surface of alumina foam samples was one of the factors that affect the low maximum stress.

4. Conclusion

During microstructure observation, a scanning electron microscope was used to observe the pore formation and pore size of the alumina foam after sintering. Based on the SEM image, it can be concluded that the single and interconnected open pores can be seen and the pores were not uniformly distributed. The pore size was in the range of 2.33 µm to 46.063 µm as the CH powder composition increased. In physical analysis, it can be confirmed that the density of alumina foam is within the range of 1.4781 g/cm^3 to 1.7936 g/cm^3 meanwhile the porosity of alumina foam is averaged 36.16% to 57.66%. It can be concluded that the higher the CH powder composition used, the lower the density of alumina foam will be. This physical analysis affects the mechanical properties of the alumina foam where the compression test was done. As the CH powder composition increased, the density decreased and porosity percentage increased, and the maximum stress that alumina foam holds decreased due to the increased pores. The alumina foam with 0 wt% CH powder composition has obtained the highest maximum stress which is 1.45853 N/mm^2 and decreased gradually to 0.35174 N/mm^2 of alumina foam with 25 wt% CH powder. The lowest CH powder composition was the best space holder composition to be used as alumina foam as it has the smallest shrinkage, highest density, smallest porosity percentage and highest maximum stress compared to others.

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