

PHYSICAL AND STRENGTH PROPERTIES OF POROUS ALUMINA PRODUCED BY SLURRY BOILING WITH DIFFERENT SOLIDS LOADINGS

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Abstract

The effect of slurry solids loading on the sintered properties of a 94% alumina foam was studied. Slurries with solids content of 30 to 45 wt% were prepared. The porous samples were fabricated using a direct foaming method by boiling the slurries until the alcohol was evaporated. Sintering of the porous alumina was conducted at 1,600 °C for 2 h. The final properties of porous alumina were investigated. It was found that the samples had similar values of apparent density from 3.55 to 3.57 g/cm³, regardless of solids loading. The apparent densities were similar because the closed pore volumes were also similar. The bulk density (1.73 g/cm³) and the porosity (52%) of samples made from slurries with solids content 30 to 40 wt% were also similar. However, the increase of solids loading to 45 wt% decreased the bulk density to 1.47 g/cm³ and increased the porosity to 59%. This was due to an increase of the open pore volume. The SEM analysis found that the 45 wt% sample had numerous small open pores, not only within the cell structure of the foam, but also in the cell walls. The flexural strengths decreased slightly from 5.5 to 4.7 MPa with increased solids loading. The results showed that the slurry solids loadings offered some control over the foam properties and that the slurry boiling method was capable of producing porous alumina with comparable properties to ceramic foams made by other methods.

Keywords: Porous alumina, Direct foaming, Slurry boiling, Solids loading

Introduction

Porous ceramics are a promising class of materials that are used in many industrial sectors. Due to their superior properties, such as high temperature resistance, low thermal conductivity, high strength, and good chemical inertness, they have been used in many applications, such as thermal insulation, heat

exchangers, filters, lightweight structural components, and catalytic converters (Şan *et al.*, 2019). The exact, suitable application for a porous ceramic depends on the structure it has. Porous ceramics with a random 3D distribution of pores are called ceramic foams. A foam is described by

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the cell structure. The cells simply being another name for the pores. An open cell foam contains a majority of porosity that is interconnected and open to the atmosphere and so has high permeability. This type of foam is useful for filtering applications. A closed cell foam contains a majority of pores that are closed to the atmosphere and so has low permeability. This type of foam is useful for thermal insulation. The strength of the foam is determined in part by the thickness of the cell or pore walls, also called struts. Defects, such as small pores, within the walls degrade the mechanical properties. An illustration of the main features of a ceramic foam is shown in Figure 1.

Ceramic foams can be fabricated by several different methods, which can be classified into three categories. They are replica templates, sacrificial fugitives, and direct foaming methods (Hammel *et al.*, 2014). Each method provides benefits and limitations. For example, a foam fabricated using a replica template can have highly interconnected pores with sizes between 200 μm and 3 mm. However, the mechanical properties are often degraded due to the pyrolysis of the organic template that leaves micropores or voids in the walls of the cells (Peng *et al.*, 2000; Studart *et al.*, 2006). The sacrificial fugitive method involves the use of a large amount of an organic pore former, up to 80%, which requires more time and energy in the subsequent pyrolysis process (Gain *et al.*, 2006). The direct foaming method tends to be the best technique compared with the other two since the processing is simpler and more environmentally friendly. The only drawback of this method is the instability of the bubbles produced during foaming. The lifetime of the bubbles must be long enough in order for the ceramic slurry to gel, and thus the addition of bonding, stabilizing, and curing agents is required in the suspension (Li *et al.*, 2014; Zhang *et al.*, 2014). These additives can add cost and complexity to the process as well potentially undesirable inorganic impurities to the resulting ceramic foams. Therefore, an alternative novel method of producing porous alumina by boiling slurry is presented in this study. This study is a continuation of our previous research (Somton *et al.*, 2018). The boiling of ethyl alcohol, i.e. ethanol, in the alumina slurry creates bubbles, foaming the slurry, which was then dried to form the porous structure. This technique is quick, simple and does not use any special curing agents, only a conventional ceramic binder system. The physical and strength properties of sintered, porous samples were examined in this paper. The pore morphologies were also observed and are discussed.

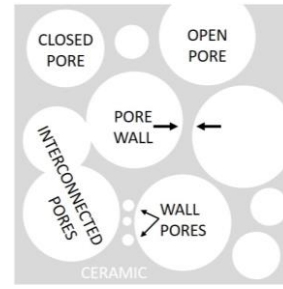


Figure 1. Illustration showing the main features of a ceramic foam applied

Methods

Alumina powder with a purity of 99.5%, and an average particle size of 4.0 μm in D[4,3], was used in the study. The powder composition for the slurries was 94 wt% alumina, 4 wt% dolomite and 2 wt% clay. The last two of which were used as sintering aids. The powders were mixed with polyvinyl butyral (PVB) at 14 wt% of the total ceramic powder mixture and stirred in ethanol for 3 min at a speed of 1,000 to 1,800 RPM to produce a slurry. Four slurries were produced with solids loadings of 30, 35, 40, and 45 wt% of the ceramic powder mixture. The slurries were poured into foil-lined paper molds and were heated using a hot plate and boiled until the ethanol was completely evaporated. The boiling point of ethanol is $\sim 75^\circ\text{C}$. After that, the foil papers were removed to obtain the green, porous samples. Sintering of the porous samples was done in air at a temperature of 1,600 $^\circ\text{C}$ with a heating rate of 5 $^\circ\text{C}/\text{min}$ and a hold time of 2 h.

For characterization, the sintered samples were cut into a bend bar shape with dimensions of 3 $\times$ 4 $\times$ 30 mm using a diamond wafering blade. The density, porosity, and pore volumes were determined using the Archimedes method. These properties were calculated using Equations 1-5 according to ASTM C-373 (ASTM, 2006). The flexural strength was measured using the 3-point bending method in a universal testing machine (UTM) with a crosshead loading rate of 0.05 mm/min. The morphology of the samples was observed by optical and scanning electron microscopy (SEM).

$$\rho_A = \frac{W_d}{W_d - W_s} \quad (1)$$

$$\rho_B = \frac{W_d}{W_w - W_s} \quad (2)$$

$$P = \frac{W_w - W_d}{W_w - W_s} \quad (3)$$

$$V_{OP} = W_w - W_d \quad (4)$$

$$V_{CP} = W_d - W_s \quad (5)$$

ρ_A	Apparent density (g/cm ³)
ρ_B	Bulk density (g/cm ³)
W_d	Dry weight (g)
W_w	Wet weight (g)
W_s	Submerged weight (g)
V_{OP}	Open pore volume (cm ³)
V_{CP}	Closed pore volume (cm ³)

Results and Discussions

The physical and strength properties of the sintered porous alumina samples are shown in Table 1. It is seen that the apparent densities of the samples made with four solids loadings were quite similar, with values from 3.55 to 3.57 g/cm³. The calculated theoretical density of the alumina with dolomite and clay sintering additives is 3.85 g/cm³. The apparent densities of the foams as a percent of theoretical density are 92 to 93%, which indicates reasonably good sintering. The bulk densities of samples made with solids loadings of 30 to 40 wt% had the same value of 1.73 g/cm³, which then decreased significantly to 1.47 g/cm³ when the solids loading was increased to 45 wt%. The porosities also had similar values of 51 to 52% for solids loadings of 30 to 40 wt% and then increased substantially to 59% when the solids loading increased to 45 wt%. These are moderate to high porosity values for a ceramic foam. The corresponding values of the open pore volumes for solids loadings of 30 to 40 wt% were 0.13 to 0.14 cm³ and increased to 0.19 cm³ when the solids loading increased to 45 wt%. The closed pore volumes for all of the solids loadings were the same value of 0.13 cm³. From these results, it can be concluded that the increase of solids loading from 30 to 40 wt% did not

significantly affect the density and porosity of the sintered porous samples. A further increase of the solid loading to 45 wt% resulted in a significant decrease of the bulk density and an increase of the porosity due to an increase of the open pore volume. This will also have created an increase of the pore interconnectivity. However, the apparent densities remained the same because of the similar closed pore volumes. This conclusion is based solely on the Archimedes calculations and so it is only useful for general discussion. Further analysis with mercury porosimetry is needed to determine the pore size, distribution and connectivity, to further clarify the results of the physical properties.

Optical microscopy images of the porous samples made with solids loadings of 30 to 45 wt% are shown in Figure 2(a-d), respectively. The samples all had a similar largest pore diameter of approximately 2 mm and showed little difference in pore size and distribution when observed with the naked eye. There was also the appearance of white and dark spots inside of the exposed pores. These were formed during the vaporization of the ethanol from the slurries. When the slurries were nearly dried, i.e. fully gelled, the increased slurry viscosity reduced the movement of the ethanol gas bubbles. Therefore, the bubbles were randomly trapped, which generated the random pore structures in the dried samples. The connected or open pores

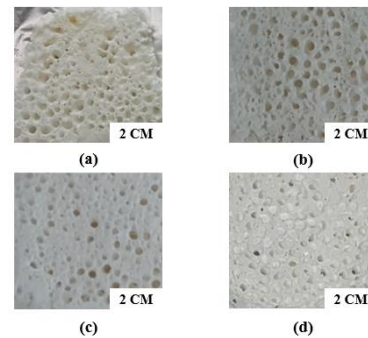


Figure 2. Optical images of the sintered samples of porous alumina obtained by slurry boiling with solids loadings of (a) 30 wt%, (b) 35 wt%, (c) 40 wt%, and (d) 45 wt%

Table 1. Measured properties of sintered specimens produced from 30 to 45 wt% slurries

Solid content (%)	Apparent Density (g/cm ³)	Bulk Density (g/cm ³)	Porosity (%)	Open pore volume (cm ³)	Closed pore volume (cm ³)	Flexural strength (MPa)
30	3.55±0.08	1.73±0.06	51±2	0.14±0.03	0.13±0.02	5.5±1.1
35	3.57±0.10	1.73±0.12	52±3	0.14±0.01	0.13±0.03	5.2±1.7
40	3.56±0.11	1.73±0.02	51±1	0.13±0.04	0.13±0.04	4.8±0.3
45	3.56±0.02	1.47±0.07	59±2	0.19±0.01	0.13±0.01	4.7±1.7

Table 2. A comparison of porous ceramic properties from several different forming methods

Porous Ceramic Research	Bulk Density (g/cm ³)	Porosity (%)	Flexural Strength (MPa)
Alumina foam (Cooymans <i>et al.</i> , 2003)	N/A	35-40	2.9
Alumina-zeolite foam (Rahman and Guan, 2007)	1.2-1.9	35-65	2.8-4.5
Porous Corundum-mullite (Chen <i>et al.</i> , 2019)	1.8-2.6	20-47	3-9
Alumina foam from this study	1.4-1.8	48-62	4.7-5.5

appeared as the black spots and the non-connected or closed pores as the white spots. The slurry with a solids loading of 45 wt% had the highest viscosity before and during the boiling so it had an increased tendency to trap bubbles. This is perhaps why the 45 wt% samples showed the largest pore volume, lowest bulk density, and the largest total porosity. SEM images of the porous morphology of a 45 wt% sample are shown in Figure 3(a-d). The large pores had a spherical shape and both open/connected (Figure 3(a)) and closed pore were observed (Figure 3(b)). There were also smaller pores distributed along and inside the larger spherical pore walls, seen in Figure 3(c). Moreover, there were also observed small, irregular shaped pores in the pore wall, seen in Figure 3(d). These images show that the porosity exists at several different size levels, including obviously the main large pores but also within the pore walls themselves.

The flexural strengths of the samples were slightly decreased from 5.5 to 4.7 MPa with increased solids loading from 30 to 45 wt%, as seen in Table 1. This would not be expected, an increased slurry solids loading is expected to increase the strength of a ceramic foam (Sarkar, 2019). The observed flexural strength trend with solids loading was possibly unclear due the large pore size in comparison to the size of the test samples. However, the measured strength values were still reasonably high. A comparison of porous ceramic properties between the direct foaming technique used in this experiment and other techniques is shown in Table 2. The porous alumina from this study has properties that are similar to those of other porous ceramics, produced by other methods.

The achieved flexural strength of 4.7 to 5.5 MPa from this study was higher than some other porous alumina-based ceramics.

Conclusions

The physical and strength properties of sintered porous alumina specimens produced by direct foaming were studied. Porous samples could be produced by boiling of alumina slurries containing

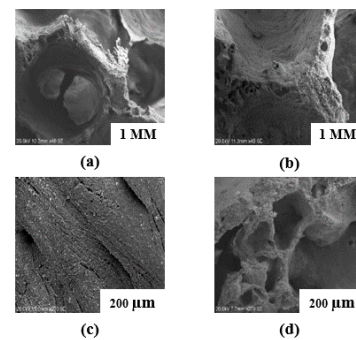


Figure 3. SEM images of the 45 wt% solids loading sample showing (a) open pores, (b) closed pores, (c) small pores distributed inside the large cells, and (d) irregular pores within the cell walls

30 to 45 wt% solids. The generated foams had millimeter scale pores with porosity in the range of 50 to 60%. The pores were a mixture of open and closed types. It was found that the solids loading of the slurry influenced the resulting physical properties of the sintered foams to some extent. An increase of solids loading lowered the bulk density and increased the porosity, due to an increase of the open pore volume. The flexural strength was slightly reduced with increased solids loading. The porous alumina produced in this study does have similar or better flexural strength compared with similar porous ceramic materials produced by other methods. These results show that with the current processing parameters, the solids loading has some effect on the resulting alumina foam properties. The results also show the promise of producing porous alumina using a direct foaming technique based on the boiling of an alumina slurry.

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