

DIFFERENT PORE SIZE ALUMINA FOAMS AND STUDY OF THEIR MECHANICAL PROPERTIES

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Recently, the open-cell ceramic foams have been extensively investigated due to special properties of these structures. They are excellent candidates for various applications such as molten metal and hot gas filtration, fabrication of metal matrix composites) MMC(, heat exchangers and catalyst support. In this study to prepare high strength and high permeable foams, alumina suspensions with proper solid contents and suitable rheological behavior were used for different pore density foams. The properties of the prepared foams such as mean pore size, total porosity, mechanical strength and water permeability were characterized by using different techniques. A reduction in pore density caused an increase in total porosity from 78.5 % to 83 %. The compression strength of the samples was dependent on total porosity as well as properties of the suspension. Compression strengths of 1.77; 3.24 and 3.55 MPa were measured for 10, 17 and 27 ppi foams, respectively. Presence of high volume of permeable pores and good uniformity of the structure led to high permeable foams. The permeability measurement confirmed a rise in permeability rate with a decrease in pore density of the foams.

INTRODUCTION

Open-cell ceramic foams have a high volume of porosity ($> 70\%$) and include a network of interconnected hollow polygonal cells [1, 2]. These structures are usually used in applications that a fluid transport is needed from a microstructure like high temperature filters for melted alloys [3, 4]. Applications such as molten metals and hot gases filtering and heat exchangers, require high permeability [4, 5]. Moreover, high mechanical strength in most structural applications is demanded as well. But, permeability and mechanical strength are inversely proportional to each other [4, 6]. The pore size and distribution and the presence of defects in the microstructure of the ceramic foams are the major factors that affect on the permeability and mechanical strength of these materials [4]. When the porosity and pore size increase in the foam, permeability increases while the mechanical strength decreases. In a given pore size and distribution, the mechanical strength of the open-cell ceramic foam can be increased by reduction of defects in the struts, while permeability does not change significantly.

The most common technique for producing open-cell ceramic foams is the replica method [7, 8]. This method involves impregnation of polymeric sponge by immersing in to a slurry containing ceramic material

and appropriate additives. After removing the excess slurry and drying, foam is heat treated to burn out of organic materials and sintering of ceramic skeleton [9]. A disadvantage of the sponge replica technique is the fact that the struts of the foam are often cracked during the pyrolysis of polymeric template [10]. Presence of triangular voids inside the struts also causes a decrease in mechanical strength.

In this work, using adequate additives in the composition and using proper solid content in the slurries, a sufficient and uniform coat is formed around the polymeric struts; therefore, the volume of defects in the struts of final produced foams has decreased significantly. The pore density control for the alumina foams was realized by the use of different pore density polyurethane sponges. The relationship between pore density, pore size, porosity, compressive strength and permeability of the alumina foams are being discussed in this article.

Experimental

Alumina Foam Preparation

In order to prepare alumina foams, α -alumina powder with mean particle size of $5\ \mu\text{m}$ was used as the main material. To improve the rheological behavior of slurry and to reduce the sintering temperature,

appropriate amounts of titanium dioxide and commercial clay were used. First the ingredients were mixed in ethanol and stirred by magnetic stirrer at a rate of 700 rpm for 3 hours. Then the mixture completely dried and was sieved through a 70 mesh screen. Poly vinyl alcohol (PVA) and Tiron were used as an organic binder and a dispersant respectively. For preparation of the slurry PVA dissolved in water, Then Tiron was added to the solution and stirred at a rate of 500 rpm for 15 minutes. Finally, ceramic powder (alumina and additives) was poured in the solution and followed a gentle stirring. To make the dispersant more effective, the slurry was aged for 48 h in a closed vessel.

The polyurethane (PU) sponges with different pore densities were immersed in the slurry and were compressed while submerging in order to fill all of the pores. The impregnated sponges were then taken out of the slurry and were pressed between two parallel plates to a specific gap to remove the excess slurry and achieve open cells and pores. The samples were aged in the air for 48 h and then were placed in an oven at 110°C for 12 h. After drying, firing of the samples took place in a programmable furnace. To burn out the organic materials without collapsing the deposited ceramic powder, the samples were heated first at a rate of 1.6°C·min⁻¹ to 600°C and then heated at 3°C·min⁻¹ to 1600°C. To complete the sintering process, the samples were kept at 1600°C for 2 h.

Characterization

The rheological behavior and viscosity of slurries were measured by Anton Paar rheometer (Physica MCR 301) in the shear rate range of 0 - 1000 s⁻¹ at 25°C. The total porosities of alumina foams were calculated by the following equation:

$$\text{Porosity} = (\rho_r - \rho_b) / \rho_r \quad (1)$$

where ρ_r is the real density of composition and ρ_b is the bulk density of the foam. Bulk density was simply calculated from the mass to bulk volume ratio of the foam. In order to determine the real density, the alumina foams were pulverized to micro sized powder. Then, density was measured by using helium pycnometer.

Compressive strength of the sintered alumina foam was measured using universal testing machine (Shirley, UK) fitted with flat steel platens closing with a cross head speed of 0.5 mm·min⁻¹. In order to provide a more uniform load distribution on the sample surface, a plastic pad was used between platens and sample. For permeability test, alumina foam with 1 × 1 × 1 cm dimension was sealed from four side in a short cylinder (see Figure 1a). Water was used as the fluid. The volumetric flow rate of water at different pressures of 0 to 7 bar was obtained by determining the volume of water that flow through the sample in 10 s under each pressure. A short cylinder with a cubic void of dimensions of 1 × 1 × 1 cm in its center

(see Figure 1b) was used as full permeable sample and volumetric flow rate of water through it was measured at pressures of 0 to 7 bar. Permeability of alumina foam was obtained from the ratio of volumetric flow rate of the foam to volumetric flow rate of full permeable sample in each pressure.

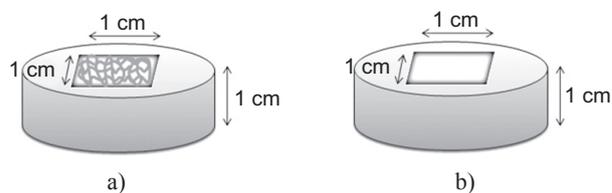


Figure 1. Schematic view of used samples for permeability test.

The cell geometry and macrostructure of the foams were observed using a digital camera (MH10, Sony, Japan). Average pore diameter of different foams was measured using stereo microscope (SZ61, Olympus, Japan) images. The microstructure of the foams was observed using scanning electron microscope (Philips, XL 30).

RESULTS AND DISCUSSION

For preparation of the ceramic foams by replica method, a slurry with special rheological behavior and viscosity is needed. Slurry should be viscose enough to more deposition on the struts of PU sponge, but the workability of high viscose slurry reduces because of difficulty enter of slurry in the sponge structure, which results in nonuniform foam. Here the shear thinning behavior and thixotropy are important as well. These properties make the slurry thin while it is under shear conditions and cause the remaining slurry that coated on the sponge network become viscose, after deletion shear stress.

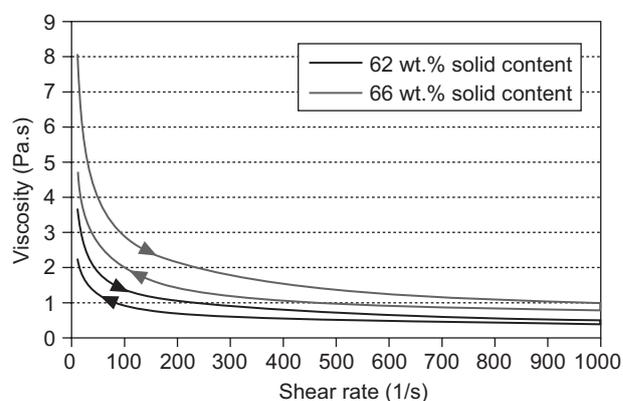


Figure 2. Viscosity curves of 62 wt. % and 66 wt. % solid content slurries.

Due to more perfect coverage on the struts, use of higher solid contents in the slurry causes to higher mechanical strength of the final foam. Because of fluid reduction and more contact of particles, an increase in solid contents in the slurry leads to higher viscosity. So, optimum solid content is necessary to have a good viscosity and final mechanical strength simultaneously.

Figure 2 shows the viscosity curves of the typical slurries that were optimized to be use in this study. Curves (a) and (b) represent 62 wt. % and 66 wt. % solid content slurry respectively. 62 wt. % solid content slurry was used for coating 23 ppi PU sponge and 66 wt. % solid content slurry was used for coating 8 and 14 ppi PU sponges. As seen in the Figure 2, viscosity decreases with increase of shear rate that indicates to shear thinning behavior. The thixotropy is also realized from hysteresis loop between the ascending and descending curves.

Figure 3 shows the sintered alumina foams with different pore densities. The foams have reticular structure and interconnected cells.

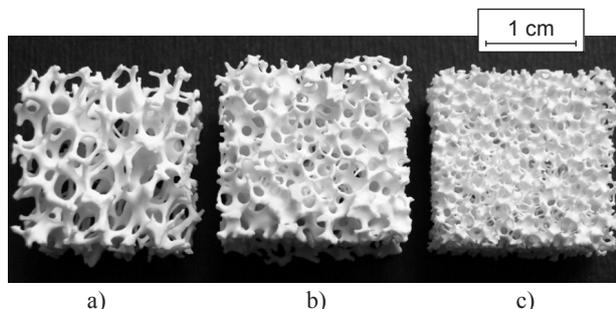


Figure 3. Morphology of alumina foams with pore density of a) 10, b) 17 and c) 27 ppi.

Upon sintering, shrinkage occurs and pore size of the foams becomes smaller. So, linear pore densities of sintered alumina foams are greater than those of polymer sponges. For example the use of a PU sponge of 8 ppi resulted in approximately 10 ppi sintered foam. Physical characterizations of alumina foams are presented in Table 1.

Table 1. The physical characterization of alumina foams with different pore densities.

Linear pore density of PU sponge (ppi)	8	14	23
Linear pore density of alumina foam (ppi)	10	17	27
Average pore size of alumina foam (mm)	1.87	1.08	0.66
Total porosity of alumina foam (vol. %)	83	80.4	78.5

As realized from Table 1, the total porosity of the alumina foams has decreased with increase of pore density. With increase of pore density, the struts become thinner (Figure 3), but the number of pores in the unit of length and so the unit of volume, increases. Up to that time when increase of pore density and reduction of pore

size do not prevent a perfect and uniform coating around the polymer struts, increase of pore density leads to more relative density and less total porosity of the foam. In fact, in this case the mass growth due to increase of pores number overcomes the mass reduction due to decrease of struts thickness.

Figure 4 shows the compressive strength of prepared alumina foams. Compressive strength has enhanced with increase of pore density that preferentially attributed to the relative density of the foams. Studies of brittle cellular materials have demonstrated a relationship between the compressive strength and relative density as follows [11]:

$$\sigma_{fc} = C \cdot \sigma_{fs} (\rho_b / \rho_r)^{3/2} \quad (2)$$

where σ_{fc} is compressive strength of the foam, C is the geometric factor, σ_{fs} is the fracture strength of dense material and (ρ_b / ρ_r) is the relative density. Hence, with increase of linear pore density the compressive strength has increased according to Equation 2.

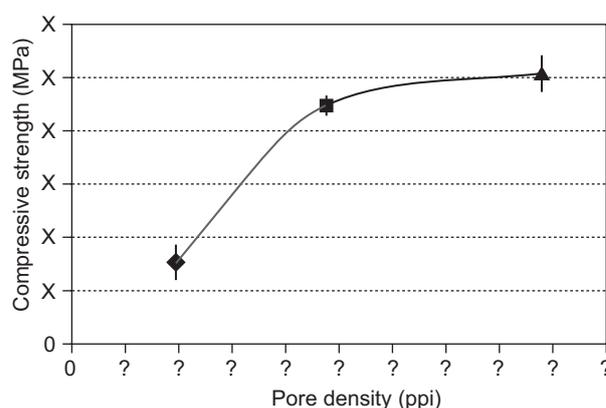


Figure 4. Compressive strength of alumina foams with different pore density.

Cell and pore size are other affecting factors on the compressive strength. With increase of pore density, diameter of cells and pores decrease and the number of struts and junctions increase in a constant volume.

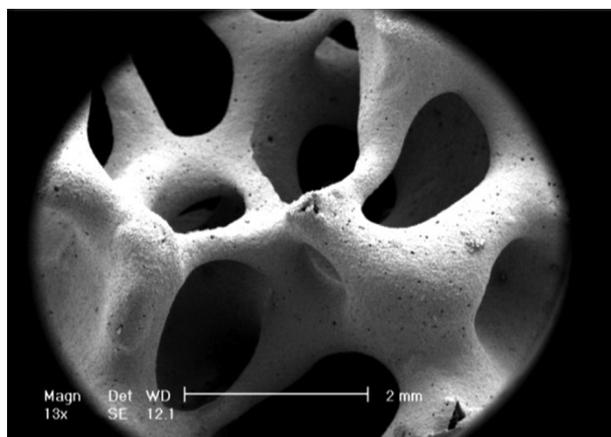


Figure 5. SEM photograph of alumina foam.

So, the load distribution in the foam network increases when a load is applied on the foam. In fact, with increase of pore density and reduction of pore size, contribution of the struts in load reception increases and this causes an increase in load capacity.

Generally, high strength values obtained for alumina foams in this research is mainly due to presence of high strength and intact struts in the foam network. Figure 5 shows the SEM photograph of 17 ppi alumina foam structure. As seen in Figure 5, struts have sufficient thickness and they are almost crack less. In addition, volume of microsize pores in struts is very low.

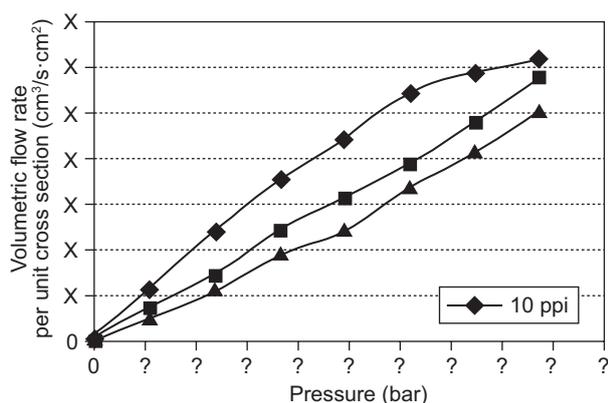


Figure 6. Volumetric flow rate of water per unit cross section of alumina foams with different pore densities.

The effect of pore density of alumina foams on the volumetric flow rate is shown in Figure 6. As seen here, with an increase in pressure and thus in the driving force, the volumetric flow rate of fluid (water) increases in all foams. In a given pressure, volumetric flow rate has decreased with increase of pore density. Various factors such as porosity, pore size and specific surface area are affected to this subject. As mentioned before, reduction of porosity leads to reduction of permeability of the foams. Also a reduction in pore size results in smaller passage for fluid permeation. In addition, with an increase in number of struts in the foam, the ratio of surface to mass becomes larger. Therefore, the fluid has to be in more contact with alumina material and this would make its flow rate decrease across the foam network.

Table 2. Water permeability of prepared alumina foams.

Linear pore density of alumina foam (ppi)	10	17	27
Permeability (%)	57	42	33

Average of the permeability measurements for each one of alumina foams are presented in Table 2. Results indicate high values of permeability for three types of alumina foams that is attributed to the presence of high volume of open and interconnected pores in their networks.

CONCLUSION

Open-cell alumina foams of different pore densities of 10, 17 and 27 ppi were successfully prepared by replica method. Rheological behavior and solid fraction of slurry have significant effect on alumina foams strength made by replication. By decrease of defects in struts, high values of strength were obtained for alumina foams, while permeability measurement confirmed high values for the foams, as well. Strength and permeability of the foams were dependent on porosity, pore density and pore size of them.

REFERENCES

1. Pryor M. J. and Gray T. J.: US Patent 3947363 (1976).
2. Scheffler M. and Colombo P.: *Cellular Ceramics: Structure, Manufacturing, Properties and Applications.*, Wiley-VCH, Weinheim 2005.
3. Řehořek L., Dlouhý I., Chlup Z.: *Ceramics-Silikáty* 53, 237 (2009).
4. Acchar W., Souza F. B. M., Ramalho E. G., Torquato W. L.: *Journal of Materials science and engineering A* 513-514, 340 (2009).
5. Fellows J. R., Anderson H. S., Cutts J. N., Lewinsohn C. A. and Wilson M. A. in: *Advances in Bioceramics and Porous Ceramics II*, Eds. R. Narayan, P. Colombo, D. Singh and J. Salem, John Wiley & Sons Inc., Hoboken USA, 2009
6. Innocentini M. D. M., Sepulveda P., Salvini V. R., Pandolfelli V. C.: *Journal of Am. Ceram. Soc.* 81, 3349 (1998).
7. Montanaro L., Jorand Y., Fantozzi G.: *Journal of Eur. Ceram. Soc.* 18, 1339, (1998).
8. Zhu X., Jiang D., Tan Sh.: *Journal of Materials letters* 51, 363, (2001).
9. Colombo P.: *Philos. Trans. Roy. Soc. A* 364, 109, (2006).
10. Studart A., Gonzenbach U.: *Journal of Am. Ceram. Soc.* 89, 1771, (2006).
11. Gibson L. J. and Ashby M.F.: *Cellular Solids: Structure and properties*, 2nd edition, p. 209-217, Cambridge Solid State Science Series, Cambridge University Press 1997.