

Porosity and cell size control in alumina foam preparation by thermo-foaming of powder dispersions in molten sucrose



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ABSTRACT

The foaming characteristics of alumina powder dispersions in molten sucrose have been studied as a function of alumina powder to sucrose weight ratio ($W_{A/S}$) and foaming temperature. The increase in foaming temperature significantly decreases the foaming and foam setting time and increases the foam volume due to an increase in the rate of $-OH$ condensation as well as a decrease in the viscosity of the dispersion. Nevertheless, the foam collapses beyond a critical foaming temperature, which depends on the $W_{A/S}$. The sintering shrinkage depends mainly on the $W_{A/S}$ and marginally on the foaming temperature. The porosity (83.4–94.6 vol.%) and cell size (0.55–1.6 mm) increase with an increase in foaming temperature (120–170 °C) and a decrease in $W_{A/S}$ (0.8–1.6). The drastic decrease in compressive strength and modulus beyond a $W_{A/S}$ of 1.2 is due to the pores generated on the cell walls and struts as a result of particle agglomeration. Gibson and Ashby plots show large deviation with respect to the model constants 'C' and 'n', especially at higher alumina powder to sucrose weight ratios.

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1. Introduction

Ceramic foams are low density materials with high specific strength, high surface area, low thermal conductivity and controlled permeability [1]. Due to this, they are used in a variety of applications such as high temperature thermal insulation, molten metal filtration, catalyst support, lightweight structural components and as a pre-form for polymer–ceramic and metal–ceramic composites [2–6]. Ceramic foams are generally prepared by the replication of polymer foams, foaming and setting of ceramic powder suspensions, and emulsion templating. In the first method, ceramic replica of a polymer foam template is produced by coating the polymer foam with a ceramic powder suspension followed by drying, burnout of the polymer foam template and sintering [7–9]. In the second method, foamed powder suspensions produced by stabilizing gas bubbles using either surfactant or particles with suitable wetting characteristic are cast in a mould [10–19]. The cast-foamed suspensions are then set by either in situ polymerization of organic monomers or by coagulation of the powder

suspensions. The wet foam bodies are subsequently dried, binder removed and sintered to produce the ceramic foams. In the third method, immiscible liquid (oil) droplets are dispersed in a ceramic precursor sol or ceramic powder suspension using a suitable emulsifying agent to form an emulsion [20–27]. The emulsion is then set by gelation of the sol or ceramic powder suspension. The gelled emulsion body is dried, oil removed and sintered to produce the macroporous ceramic foams. Use of liquid droplet as pore template is advantageous, as a uniform dispersion of the immiscible liquid droplets in an aqueous slurry medium using an emulsifying agent could be obtained by simple stirring. Also, the pore size could be manipulated by controlling the droplet size, which can be obtained by adjusting the emulsifying agent concentration and mixing speed. High internal phase emulsions [24,27,28] with liquid template phase more than 74 vol.% are possible which leads to macroporous ceramics with very high porosity and highly interconnected pore structure.

Recently, we have reported a thermo-foaming of powder dispersions in molten sucrose for the preparation of alumina foams [29]. In this, alumina powder dispersions in molten sucrose were thermo-foamed at 150 °C to produce a sucrose polymer–alumina powder composite foam bodies. Subsequent sucrose polymer burnout and sintering produces well interconnected cellular ceramics with high porosity and relatively high compressive strength. The method is simple and does not use any toxic chemicals

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as processing additives. Moreover, large sintered alumina ceramic bodies without any crack could be fabricated by this process by introducing an intermediate pyrolysis of the sucrose polymer before burnout and sintering [30]. Moreover, we have observed a slight increase in sintering shrinkage, decrease in porosity and increase in compressive strength by the intermediate pyrolysis of the sucrose polymer. However, the alumina foams produced by thermo-foaming at 150 °C from dispersions of alumina powder to sucrose weight ratios ($W_{A/S}$) in the range of 0.6–1.4 showed porosities in a close range of 93.3–94% [29]. The present work is an effort to control the porosity and cell size of the alumina foams to a wide range by controlling the foaming temperature as well as the $W_{A/S}$. The study also provides the effect of temperature and $W_{A/S}$ on the foaming time and foam setting time of alumina powder dispersions in molten sucrose. In the present work, the $W_{A/S}$ of 0.8 and above is used, as cracks were often formed during sintering of the foam bodies prepared at a $W_{A/S}$ below 0.8 [30].

2. Experimental

α -Alumina powder (A16SG, ACC Alcoa, Kolkata) of 0.34 μm average particle size and specific surface area of 10.4 m^2/g was used. Analytical reagent grade sucrose and acetone used were procured from Merck India Ltd., Mumbai. The procedure for thermo-foaming of alumina powder dispersions in molten sucrose reported in our previous publication was adopted without any modification [29]. The sucrose (200 g) and alumina powder in various weight ratios were intimately mixed by planetary ball milling (Fritsch, Germany) at 200 rpm in acetone medium for 4 h using zirconia grinding media of 10 mm diameter in 500 ml zirconia jars. The slurries thus obtained were dried in glass trays at 80 °C in an air oven. The alumina powder–sucrose mixtures obtained after drying had $W_{A/S}$ in the range of 0.8–1. This corresponds to volume percentage of alumina in the mixtures in the range of 24.3–41.8 (density of sucrose – 1.59 g/cm^3). The sucrose–alumina powder mixtures were heated in 2.5 l borosilicate glass trays at 185 °C in an air oven to melt the sucrose. The melt was stirred well with a glass rod to get uniform dispersion of the alumina powder in the molten sucrose. The alumina powder dispersions in the molten sucrose were kept in an air oven at temperatures ranging from 120 to 180 °C for foaming and setting. The foams were cut into rectangular bodies of 10 cm \times 10 cm \times 4 cm and heated in an inert (Argon) atmosphere furnace at 900 °C for 2 h for pyrolysis of the sucrose polymer [30]. The heating rate used was 0.5 °C/min. The pyrolyzed foam bodies were heated in an electrically heated sintering furnace in air atmosphere up to 1600 °C for removal of the carbon and subsequent sintering. The heating rates used were 0.5 °C/min and 1 °C/min from room temperature to 600 °C and from 600 °C to 1600 °C, respectively. A holding time of 2 h was given at 600 °C and 1600 °C. The volume shrinkage of the bodies was calculated from the initial and final dimensions. The density of the alumina foam bodies was calculated from their weights and dimensions.

The viscosity measurement of the alumina powder dispersions in molten sucrose was carried out at various shear rates at temperatures ranging from 120 to 160 °C using a rheometer (MCR 102 Modular Compact Rheometer, Anton Paar, USA) with a cone and plate measurement system (CP-25, 25 mm diameter and angle 2°). The microstructure of the foams was observed using a scanning electron microscope (SEM, FEI Quanta FEG200). The cell size was measured on the magnified image of the alumina foams observed using a vision inspection system with a CCD colour camera (Vision 300 GL, TESA Technologies, Switzerland). The values reported were average of 25 measurements. The compressive strength of the alumina foams was measured according to the ASTM standard C365/C365-05 using rectangular samples

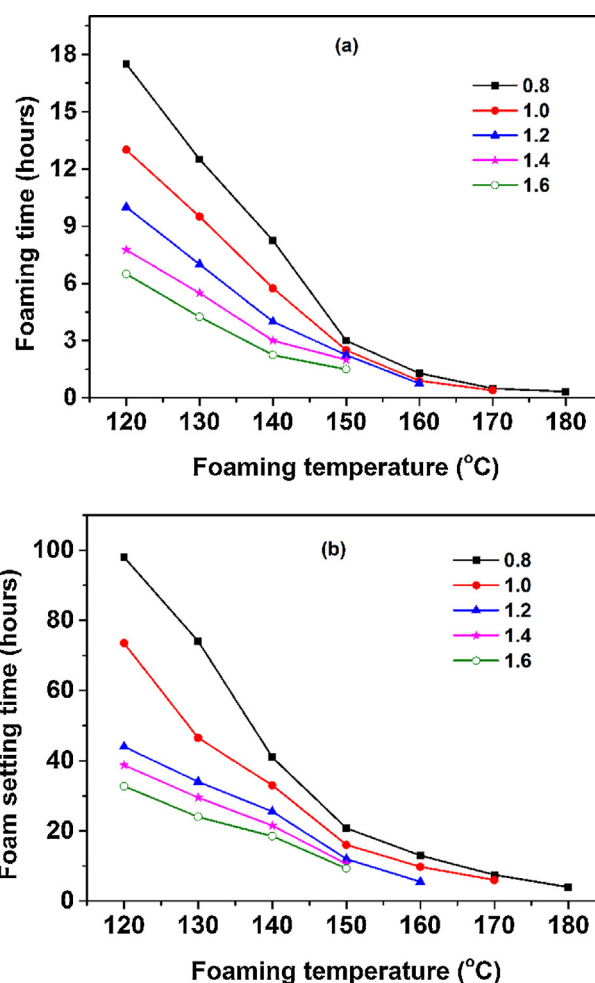


Fig. 1. Effect of foaming temperature on (a) foaming time and (b) foam setting time of dispersion at various $W_{A/S}$.

of 25 mm \times 25 mm \times 12 mm size in a Universal Testing Machine (Instron 5500, Instron USA) at a loading rate of 0.5 mm/min.

3. Results and discussion

It has been reported that the foaming and setting of alumina powder dispersions in molten sucrose is due to the –OH to –OH condensation between the glucose and fructose anhydride molecules formed from the sucrose [29]. That is, water vapour generated due to the condensation reaction is responsible for the foaming of the alumina powder dispersions. The bubble stabilization and setting of the foamed dispersions is due to viscosity increase by the continued condensation polymerization. The alumina particles in the dispersion promote foaming and foam setting by facilitating the –OH condensation. Further, the alumina particles adsorbed on the liquid–air interface offer additional bubble stability by the particle stabilization mechanism. Fig. 1 shows the effect of temperature on foaming and setting of alumina powder dispersions of various $W_{A/S}$. At all the studied $W_{A/S}$, the foaming time and foam setting time decrease significantly with an increase in foaming temperature. For example, the foaming time at a $W_{A/S}$ of 0.8 decreases from 17.5 to 0.5 h when the foaming temperature increases from 120 to 170 °C. The corresponding decrease in foam setting time is from 98 to 7.5 h. On the other hand, at a particular foaming temperature, the foaming time and foam setting time increase with a decrease of $W_{A/S}$. The difference observed in foaming time and foam setting time between powder dispersions of

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