Improvement of Microstructural Controllability of Cellular Ceramic for Multifunctional Composites

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Abstract. By modifying the sequences in preparation of aqueous ceramic slurries, starch was successfully employed to improve the microstructural controllability of alumina foams fabricated through protein coagulation casting technique. Open porosity was increased and the uniform distribution of microstructures was improved by the dual functions of starch as pore former and wet foam stabilizer. The viscosity of slurry was affected by the increasing amount of starch additive due to the total solid loading increase and the water uptake of starch. A well controlled pore structure will benefit multifunctional applications of these porous foams.

Introduction

Porous ceramics have been widely used as filters, catalysts, thermal insulations and high temperature combustion burners, composites and bio-materials [1-2]. Numerous processing routes have been extensively researched and reviewed for the production of porous ceramics with specific applications [3-4]. In the recently developed Protein Coagulation Casting (PCC) method, protein-based aqueous slurry was used to fabricate the ceramic and metal foams through direct foaming and casting [5-6]. Due to the nature of the protein foaming mechanism in PCC technique, it is very challenging to increase both the total porosity and the open porosity of ceramic foams, especially with the slurries having relatively high viscosity [7]. On the other hand, it was demonstrated that the addition of pore former increased the open porosity of prepared ceramic foams, but the distribution of pores became non-uniform as a result of the increasing viscosity [8]. However, the applications of ceramic foams in both bi-continuous composites and bio-implants require the inter-connective microstructures which enable the liquid infiltration in the former case and facilitate the cellular growth, nutrition transportation and waste drainage in the latter case. Also, the uniform distribution of microstructure is essential to the functional and structural properties of the porous ceramics. In the present work, a combination of environmentally acceptable slurry compositions, low-cost processing method and a resourceful natural material, i.e. the PCC and starch, is investigated with the aim to improve the microstructural controllability of ceramic foams.

Experimental details

Alumina powder (Alcoa CT3000SG), poly ammonium salt dispersant (Duramax D-3005, Rohm and Haas, Philadelphia, PA), egg albumen powder (Sanovo Egg Product U.K. Limited) and

potato starch (Fisher Scientific, U.K.) were selected for this study. The detailed compositions of the ceramic slurries can be found elsewhere [7]. The procedure for fabricating the ceramic foam is depicted in Fig. 1. Viscosity of the alumina slurry was measured on a coaxial cylinder rheometer (Bohlin Gemini, Malvern Instruments Ltd. U.K.) with a parallel plate configuration (40 mm diameter) and 1 mm gap perform with various shear rates ranging from 0.1 to 100 s⁻¹ at 25°C. The total and open porosity of alumina foams were measured by following the ASTM standard C20. The microstructure of the foams was observed by using scanning electron microscopy (JEOL JSM5600LV) and images were analyzed by Image J, 1.33u.

Fig.1 Flow chart of the fabrication of alumina foam.

Fig.2. Effect of addition of starch to the open porosity of alumina foam (see text for sample code).

Results and discussion

The microstructures of the resulting alumina foams consisted of spherical hollow cells, which can be commonly found in porous materials prepared via direct foaming methods. Pore distribution tends to be more uniform at relatively lower solid loadings. With increasing alumina loading, window size of the pores decreased and the pores tend to be closed. The initial pore size is proportional to the amount of air trapped inside during the tumbling, while the ultimate pore size of the porous ceramic depends on a balance between the kinetics of bubble destabilization and the speed of suspension setting. Increasing the viscosity of slurry can directly hinder the incorporation of gas, which in turn decreases the bubble size, hence leads to a remarkable decrease of total porosity and pore size. The porosity of the alumina foam with and without addition of starch was compared in Fig.2, in which the samples were denoted by using letter 'A' and 'S' for alumina loading and amount of added starch used in slurry, respectively. It is interesting that the addition of starch had no apparent effect on the total porosity of ceramic foams, except for the sample A55S10, whose total porosity increased from 49% to 57.31%, while the open porosity of all samples were significantly increased. Ceramic foams with 30 vol%, 35 vol% and 45 vol% alumina loadings are almost completely open-porous. The increase in open porosity is particularly significant for samples made from high solid loading slurries as open porosity nearly doubled, which is mainly resulted from the added starch to increase the interconnectivity between neighbouring cells (Fig.3).

Fig.3 Apparent viscosities at a shear rate of 0.5 s^{-1} of slurries with various solid loading

Fig.4. SEM micrograph shows the uniform microstructure of ceramic foams fabricated from slurry with 45 Vol% alumina but different weight ratio of starch to alumina (a) 1:10(b) 1:20

The viscosity of slurry was affected by the combined functions of increasing total solid loading and water uptaking of starch. It has been demonstrated that viscosity played a dominant role in controlling the microstructures of ceramic foam in the PCC processing technique [7-8]. However, starch noticeably not only acts as an organic fugitive in green body to increase the open porosity, but also as a wet foam stabilizer to affect the microstructure of alumina foam, such as the distribution of pore size. As illustrated in Fig. 6, at the same alumina loadings of 45 vol%, the alumina foams showed very uniform microstructures, even when the starch to alumina weight ratio was doubled from 1:10 to 1:20. This is in contrast to the observations reported in the literature [8] that a higher content of starch resulted in a non-uniform pore distribution. The uniform microstructure obtained in the present work is likely due to the different processing procedure: instead of mixing the egg white powder, alumina particles and starch with water simultaneously as reported in literature [8], the present ceramic slurry was obtained by premixing aqueous egg white solutions with alumina powder and starch was then added to the prepared solid suspension. This modified procedure helped achieve more uniform distribution of alumina in slurry, as alumina particles were well dispersed before the addition of starch.

From multifunctional application perspectives, a bi-continuous metal-ceramic composite was successfully fabricated by infiltrating the alumina foams prepared in this study with molten aluminium (Fig.5), the resulting microstructure possesses a good combination of stiffness and conductivity which will be discussed in detail elsewhere [9].

Summary

The starch additive not only acts as a fugitive phase to increase the interconnectivity of pores, but also affects the size and the distribution of pores in the resulting ceramic foams. By adding the starch powder into premixed ceramic slurries in which the ceramic particles were already well dispersed, the microstructures of the prepared foams are found to be uniform even at relatively high starch loadings. The modified procedure of introducing starch into the slurry systems provides better microstructural controllability in the PCC technology. This is beneficial to the multifunctional application of these ceramic foams.

Fig.5. SEM image of a bi-continuous composite in which dark phase is reinforcing Al₂O₃ foam and light phase is Al matrix.

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