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ScienceDirect

Ceramics International 41 (2015) 2599–2606

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Short communication

Mechanical properties and thermal conductivity of porous alumina ceramics obtained from particle stabilized foams

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Received 20 August 2014; received in revised form 22 September 2014; accepted 1 October 2014
Available online 13 October 2014

Abstract

In this work, foams were obtained by direct foaming of aqueous alumina suspensions containing butyric acid. Butyric acid is incorporated in the suspensions in order to hydrophobize alumina particles, leading to their adsorption at the air/water interface. After setting and drying, wet foams were sintered at 1585 °C for 2 h. The resulting porous samples were characterized in terms of microstructure, mechanical properties and thermal conductivity. Microstructures of sintered alumina foams reveal closed pores surrounded by a thin alumina layer. The microstructure of these macroporous ceramics is related to (i) the composition of the initial suspension (alumina and butyric acid contents) and (ii) the stirring velocity during the foaming process. Macroporous ceramics with pore sizes (d_{50}) ranging from 20 μm to 140 μm and porosities between 25% and 89% were obtained. Three-point bending tests were performed on foams with porosities between 65% and 89%. The results of mechanical tests were analyzed with Weibull statistic. The bending strength values are between 5 MPa and 42 MPa. The Young's modulus, obtained from 3-point bending tests, decreases with the porosity level according to Gibson–Ashby model. Thermal diffusivity measurements were made with the laser flash technique in order to determine the effective thermal conductivity. Experimental values are significantly higher than the predictions with Landauer's relation and almost close to Maxwell/Hashin–Shtrikman upper bound.

Keywords: B. Porosity; C. Mechanical properties; C. Thermal conductivity; D. Al_2O_3; Foams

1. Introduction

Porous ceramics offer interesting properties in comparison with dense ceramics: permeability to fluids, high specific surface area, low density, low thermal conductivity, thermal stability, high corrosion and wear resistance, etc. But the above mentioned properties are related to the microstructure of the porous materials. Open pores are required for applications as filters, carriers for catalysts, bioreactors and bone substitute. On the other hand, closed pores are useful for sonic and thermal insulations or for lightweight components [1].

There are several methods to produce macroporous ceramics: (i) partial sintering of powder compact, (ii) replica technique, (iii) sacrificial templating and (iv) direct foaming [2,3].

In the present work, macroporous ceramics were obtained from particle-stabilized wet foams, according to the procedure described by Gonzenbach et al. [4–6]. In this method, short-chain amphiphilic molecules are added to the ceramic suspensions before foaming in order to hydrophobize particles, leading to their adsorption at the air/water interface.

This study focuses on the preparation and characterization of alumina foams. Two parameters affecting the microstructure of the porous samples were first investigated: (i) the composition of the initial suspension (alumina and butyric acid contents) and (ii) the stirring velocity during the foaming process. Then, the mechanical properties of porous ceramics were evaluated with 3-point bending tests. The stress at break and Weibull

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http://dx.doi.org/10.1016/j.ceramint.2014.10.011
0272-8842/

moduli were obtained. The influence of the porosity on the Young's modulus was discussed through the comparison between the measured values and the predicted one's. The analytical models chosen for Young's modulus prediction are the Hashin–Shtrikman upper bound and the Gibson–Ashby expression. Finally the effective thermal conductivity of alumina foams was determined and related to their particular microstructure. Experimental values were compared with Maxwell/Hashin–Shtrikman upper bound and Landauer's expressions to investigate the influence of the porosity on the effective thermal conductivity.

2. Materials and methods

2.1. Samples preparation

Suspensions were prepared by adding α-alumina particles (Alcan P172 LSB, d_{50}=0.41 μm) to deionized water. The solid content was initially set to 41 vol%. The pH of the suspensions was fixed at about 5 using a 2 M HCl solution in order to electrostatically stabilize alumina particles. The deagglomeration of the suspensions was performed using a Turbula mixer for about 1 h. Butyric acid was then added to the alumina suspensions. Subsequently the pH of the suspension was adjusted to 5 by adding 2 M NaOH solution. Finally the amount of water needed to adjust the solid content was added.

Once the suspensions with the desired solids and butyric acid contents have been obtained, foams can be prepared. Bubbles are incorporated in the suspensions (150 ml) using a household mixer equipped with two whisks. Two stirring velocities were tested in order to identify the influence of this parameter on the microstructure of sintered samples: 220 and 300 revolutions per minute (rpm) for 2 min.

The foamed suspension was then poured into silicone molds with dimensions 80 mm × 80 mm × 8 mm.

Wet foams were first dried for 48 h at ambient temperature under high relative humidity (about 90%). Another drying period of 48 h is required under ambient humidity and temperature conditions.

Sintering was performed in an electrical furnace (HT 16/17, Nabertherm) at 1585 °C for 2 h. Heating rate was set to 1 °C/min.

2.2. Characterization of macroporous ceramics

2.2.1. Analysis of the microstructure

The porosity values v_p of the sintered samples were obtained from the bulk and true densities using the following relation:

$$v_p = 1 - \left(\frac{\rho_{\text{bulk}}}{\rho_{\text{true}}}\right) \tag{1}$$

The bulk density (ρ_{bulk}) was determined from the mass and geometrical dimensions of the samples. The true density (ρ_{true}) of alumina is 3.98 g/cm^3.

Microstructures of the sintered samples were observed using a JEOL JSM-5900 LV scanning electron microscope in SEI mode. The pore sizes were measured with "Mesurim" software®. The d_{10}, d_{50} and d_{90} diameters were obtained from the cumulative number distribution meaning that x% of the pores have a smaller diameter than d_x.

Microstructure, pore size distribution and porosity level were related to the composition and the rheological behaviour of the alumina suspensions. Rheological measurements were carried out using a controlled rheometer (Haake RS 150, Karlsruhe, Germany) with coaxial cylinder geometry (Z41).

2.2.2. Mechanical tests

3-point bending tests were performed using an INSTRON universal testing machine. The sintered samples were cut in the shape of 3 mm × 4 mm × 45 mm parallelepipeds to satisfy the geometry requirements of EN 843-1 standard. All measurements were carried out at a load rate of 0.5 mm/min.

The bending strength σ_f is given by Eq. (2):

$$\sigma_f = \frac{3\,P\,L}{2\,w\,t^2}, \tag{2}$$

where P is the fracture load (N), L is the support span (40 mm), w is the width of the sample (4 mm) and t is its thickness (3 mm).

Young's modulus E was calculated according to the following expressions:

$$E = \frac{P\,L^3}{48\,y\,I}, \tag{3}$$

$$I = \frac{w\,t^3}{12}, \tag{4}$$

where y is the deflection (mm) and I is the moment of inertia of the cross-sectional area.

The results of mechanical tests were analyzed by Weibull statistical theory. The Weibull modulus m is a measure of the scatter of the data and corresponds to the slope of the plot ln (ln(1/(1−F)) versus ln σ_f. F is the failure probability, n/(N+1), with n the ranking of the sample and N the total number of samples.

2.2.3. Thermal conductivity measurements

The thermal conductivity (λ) is obtained with the following expression:

$$\lambda = \alpha\,\rho\,c, \tag{5}$$

where α is the thermal diffusivity, ρ is the bulk density and c is the specific heat of the material.

Thermal diffusivity measurements were performed on disk samples (1.5 mm in thickness and 10 mm in diameter), at ambient temperature (23 °C), with the laser flash technique. The laser is used to heat up the front face of the disk sample. The absorbed heat diffuses throughout the sample and the back face temperature is monitored with an infrared detector (Hg–Cd–Te). Samples were coated with a thin graphite layer from a spray can in order to avoid the transmission of the laser radiation through the thickness of the material and to improve the laser beam absorption and the emitted signal of the back face. Degiovanni's method, which takes into account heat losses, was used to calculate the thermal diffusivity from the analysis of the back face temperature-time behaviour [7].