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Preparation of ultralight glass foams via vacuum-assisted foaming

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ABSTRACT

We report a novel vacuum-assisted foaming technique for preparing ultralight glass foams using green spheres as raw material. The green spheres were sintered to form ultralight glass foams under vacuum pressure. The effect of sintering temperature and vacuum pressure on the preparation and properties of glass foams was investigated. The porosity and cell size increased with the decrease of vacuum pressure. Glass foams were fabricated at vacuum pressure of 30–70 kPa, with bulk density, porosity and compressive strength values of 0.081–0.122 g/cm^3, 95.2–96.8% and 0.29–1.18 MPa, respectively. The ultralight glass foams have potential applications as thermal and acoustic insulation materials.

1. Introduction

Glass foam is a heterophase system consisting of the solid and gaseous phase. As possessing properties including lightweight, thermally insulating, sound absorbing, compression-resistant and nonflammable, glass foam has been extensively applied in many fields such as lightweight ceramics, catalyst supports, structural materials, thermal and acoustic insulation materials [1–3]. Recently, preparation of glass foams using waste glass has raised a great practical interest due to the environmental pollution [4–7]. The most common method is to sinter waste glass powder admixed with suitable blowing agents. The blowing agents produce gaseous products during the sintering process, which may be caused by an oxidation effect of C-based blowing agents (carbon black, organic compounds, SiC, etc.) or by a decomposition process of minerals ($MgCO_3$, $CaCO_3$, etc.) [8–9]. In addition, glass foams can be synthesized from waste glasses using a facile green chemistry route that involves a low-temperature hydrothermal ion-exchange reaction [10]. While considerable progress has been made on the fabrication of glass foams from waste glass, glass foams with higher porosity and mechanical strength are highly desirable.

Basically, a reduction of the pressure would increase the size of already existing pores keeping the surrounding parameters constant, according to the gas equation

$$PV = nRT \quad (1)$$

where P, V and n describe the gas pressure, volume and amount of gas, respectively. T is the temperature and R is the gas constant, which is 8.314 J/mol K. Based on this principle, the vacuum foaming technique has been used for the fabrication of magnesium foams [11]. And the vacuum-assisted foaming technique has been successfully used for foaming of ceramic suspensions [12–13]. Furthermore, high strength borosilicate foams have been fabricated by melting glass powder under high-pressure argon gas and subsequent heat treatment of the glass bulk at atmospheric pressure [14]. However, few attentions have been focused on preparation of glass foams using this superior method.

Herein, we demonstrate a novel vacuum-assisted foaming process for the fabrication of ultralight glass foams using green spheres as raw materials. Vacuum condition enhances the growth of bubbles or cells during the foaming process. In this paper, the effects of sintering temperature, sintering procedure and vacuum pressure were investigated.

2. Experimental procedure

Green spheres (Hebei YL-Bangda New Materials Limited Company, Handan, China), with size distribution of 30–200 μm, were used as raw material. The green spheres were prepared by the spray drying of particle-stabilized foam slurry composed of recycled soda-lime glass powder, dispersants (sodium hexametaphosphate), binders (polyvinyl alcohol), water and foaming agents

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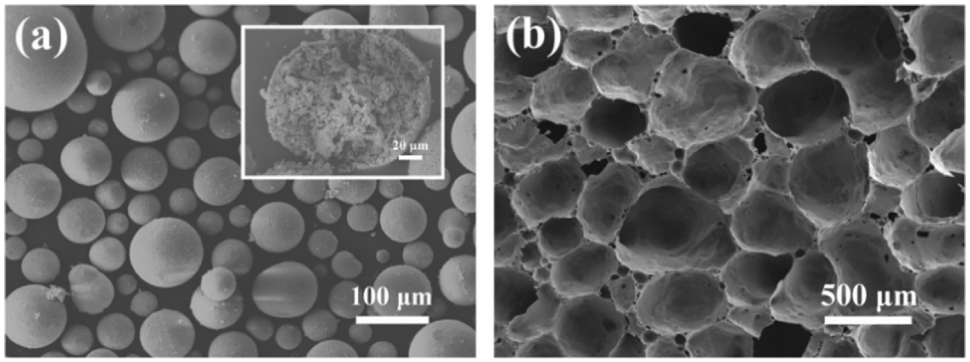

Fig. 1. SEM images of (a) green spheres and (b) the glass foam sintered at 700 °C, the inset in (a) shows the inner microstructure of a single green sphere.

(propyl gallate) [15]. As shown in Fig. 1(a), the spheres are porous with many small holes between glass particles in the interior.

The green spheres were stacked in porcelain crucibles coated with a thin layer of kaolin on the internal surface. The samples were heated up to the sintering temperature range between 660 °C and 720 °C at a rate of 3 °C/min and then held for 2 h. Different sintering procedure and vacuum pressure were used to prepare samples. The whole process of pumping air into vacuum state was finished in less than a minute.

The microstructures of green spheres and samples were investigated using scanning electron microscope (Merlin VP Compact, Zeiss, Germany). Compressive strength was measured using a universal material testing machine (AG-IC 20 kN/50 kN, Shimadzu, Japan) at a loading rate of 1 mm/min. The carbon content measurements were performed using an elemental analyzer (EA3000, EuroVector, Italy). The mean cell diameter was determined by analyzing images using graphics software. The bulk density, ρ_b, was calculated with mass and volume of the test sample with regular shape. The total porosity, P, was determined with the following equation:

$$P = (1 - \frac{\rho_b}{\rho_r}) \times 100\% \quad (2)$$

Where ρ_r (ρ_r=2.53 g/cm^3) was the real density of glass powder. The volumetric water absorption, W_v, was calculated using Eq. (3):

$$W_v = \frac{m_1 - m_0}{\rho_w \times V} \times 100\% \quad (3)$$

where m_0 and V were the mass and volume of the test sample. ρ_w was the density of water at room temperature. m_1 was the mass of the test sample immersed in deionized water for 24 h at room temperature.

3. Results and discussion

3.1. Effect of sintering temperature

The properties of samples are illustrated in Table 1. Samples were sintered at atmospheric pressure. Green spheres play an important role during the foaming process. Each green sphere forms a separate space for foaming and generates small and uniform cells. Another unique advantage of using green spheres is that the spheres can prevent gaseous products from escaping. Table 2 shows the carbon contents of different samples. The carbon content decreases from 1.34 wt% to 0.48 wt% when the sample is heated up to 700 °C, which indicates the fact that the organic compounds used in the preparation process of green spheres transform into carbonaceous products on incomplete pyrolysis of the polymers during the heating procedure. And mixtures of CO_2 and CO are commonly produced by the oxidation of carbonaceous products [8]. The carbon content decreases to 0.33 wt% after the foaming process at 700 °C. With the increase of sintering temperature, the gaseous products produced during the sintering process increase, contributing to the decrease of bulk density. The volumetric water absorption is less than 4.0% between 660 °C and 700 °C. However, the volumetric water absorption increases sharply at temperatures higher than 700 °C, which is attributed to the open-celled microstructure. The sample sintered at 700 °C shows good integrated performance, with a uniform microstructure as shown in Fig. 1(b).

Table 1
The properties of samples sintered at different temperatures.

Sintering temperature (°C)	660	680	690	700	720
Bulk density (g/cm^3)	0.257	0.180	0.157	0.134	0.128
Volumetric water absorption (%)	1.3	1.9	1.4	3.6	9.3

Table 2
The carbon contents of different samples.

The sample	Carbon content (wt%)
Green spheres	1.34
Green spheres heated up to 700 °C	0.48
Sample sintered at 700 °C	0.33

3.2. Effect of sintering procedure

The schematic diagram of sintering procedure is shown in Fig. 2 (a). The sintering temperature is 700 °C and the vacuum pressure is 50 kPa. T_n is the temperature at which air in the furnace is pumped into vacuum. Fig. 2(b) shows the effect of T_n value on microstructures and properties of glass foams. The bulk density decreases as T_n value increases, while the cell size is a positive function of T_n value. With the increase of T_n value, more gaseous products are enclosed in the cells before the vacuum condition, resulting in the increase of cell size and the decrease of bulk density. Another important reason for the increase of cell size is the coalescence of cells. The photographs in Fig. 2(b) demonstrate that the coalescence of cells ultimately leads to the uneven microstructure of the foams. The volumetric water absorption has great dependence on the open cells existing in the samples. The sample with the T_n value of 700 °C has higher volumetric water absorption, which is attributed to the open cells resulting from the coalescence of cells.