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Fabrication of Porous Ceramics by Direct Foaming

THE AUTHOR

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ABSTRACT

Porous ceramics have been extensively studied during the last two decades because of their application potentials in various fields including thermal insulators, radome materials, gas or molten metal filters, catalytic supports and biomedical substitutes for bone. This paper gives a brief review on the recent developments of preparation of porous ceramics by direct foaming method, it shows that the direct foaming method is a more effective way for preparation of high performance porous ceramics with high porosity, high mechanical strength and an even pore size distribution compared with conventional methods.

KEYWORDS

porous ceramics, direct foaming, preparation, mechanical properties
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1 Introduction

Porous ceramics have attracted more and more attention due to their excellent combined properties such as high porosity, high surface area, high permeability, good thermal shock resistance, low dielectric constant, low thermal conductivity, and their light weight and high damage tolerance. In general, it is difficult to achieve these properties from their conventional dense counterparts, and thus, porous ceramics are considered as candidates for thermal insulators, gas filters, catalytic supports, separation membranes, high-temperature structural materials, kiln furniture, radome materials and biomedical substitutes for bone [1–10]. During the past years, many methods have been developed to prepare porous ceramics, such as partial sintering [11, 12], replica [13–15], sacrificial templates [3,16–19], direct foaming [20, 21], freeze casting [22, 23] and three-dimensional printing [9, 24]. Among these methods, direct foaming is an attractive process for the fabrication of porous ceramics because it is simple, convenient, eco-friendly and cost-effective. Moreover, the route can be also used to prepare porous ceramics with complex shape, controlled pore size and the desired mechanical properties.

In the direct foaming process, bubbles that are usually generated in ceramic slurries or in the polymeric precursor solution to create a stable foam structure are very important for the preparation of porous ceramics, and these bubbles can be produced via chemical or physical processes, such as bubbling gas into slurry and mechanical mixing by using surfactants as the foaming reagent [25–28]. However, the problem with the process is that these bubbles are likely to coalesce and then result in large pores in porous ceramics due to thermodynamic instability. Thus, the most critical factor for the direct foaming process is foam stabilization, which plays an important role in the performance of the final porous ceramics. To avoid the collapse of the foam and to increase foam stabilization, it is of great significance to add some special additives into the foamed slurry to form a porous ceramic body with high strength. In this article, we provide an overview of the recent research on the preparation of porous ceramics by the direct foaming method using various ways to solidify the foamed slurry.

2 Temperature-induced gelling process

An approach relying on the temperature-induced gelling of natural macro-molecule compounds such as agarose [29], sucrose [30], agar [31], egg yolk or egg white [32–35], gelatine [36] and starch [37–39] was recently applied as non-toxic gelling reagent for the solidification of foamed slurry.

Potoczek [29] manufactured alumina foams with porosity ranging of 86–90 %, pore size ranged from 529 to 375 µm, average window size varied from 77 to 113 µm, flexural strengths between 2.71 and 5.50 MPa and compressive strengths in the range of 4.01–8.18 MPa, using agarose as the temperature-induced gelling reagent. Figure 1 shows the SEM cross-section of the prepared porous alumina ceramics with various amounts of agarose; it indicates that the porous alumina ceramics had approximately spherical cells interconnected by circular windows, and the cell size and the window size decreased with increasing agarose concentration in the starting slurry. Fadli's group [32,33] have developed a new protein foaming-consolidation method to prepare porous alumina and porous alumina-hydroxyapatite (HA) composites using egg yolk both as consolidating and foaming reagent. The compressive strength of the porous alumina ceramics with pore sizes of 25–1000 µm and a relative density of

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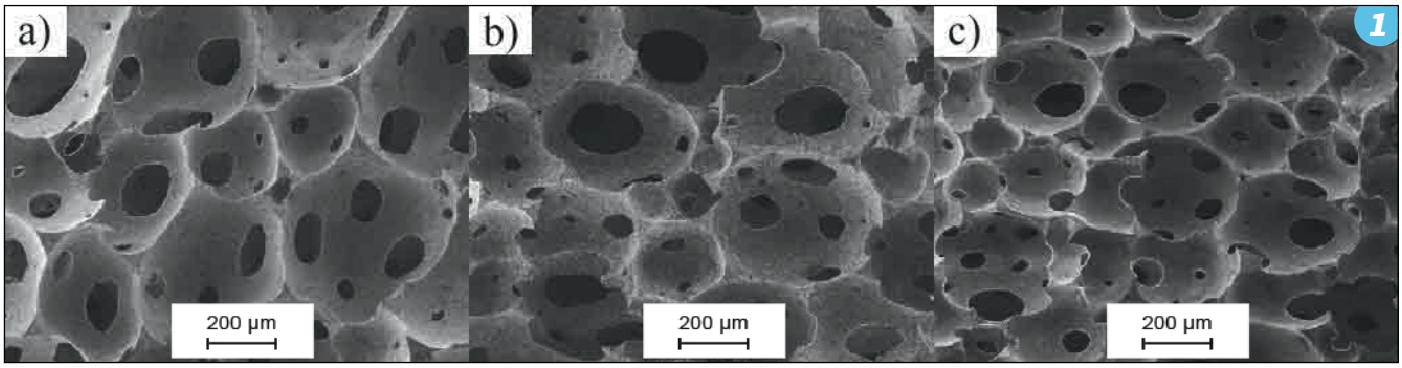

Fig. 1 • SEM cross-section of alumina foams prepared with 35 vol.-% slurry and agarose contents of: a) 0.50 mass-%, b) 0.75 mass-%, c) 1.0 mass-% [29]

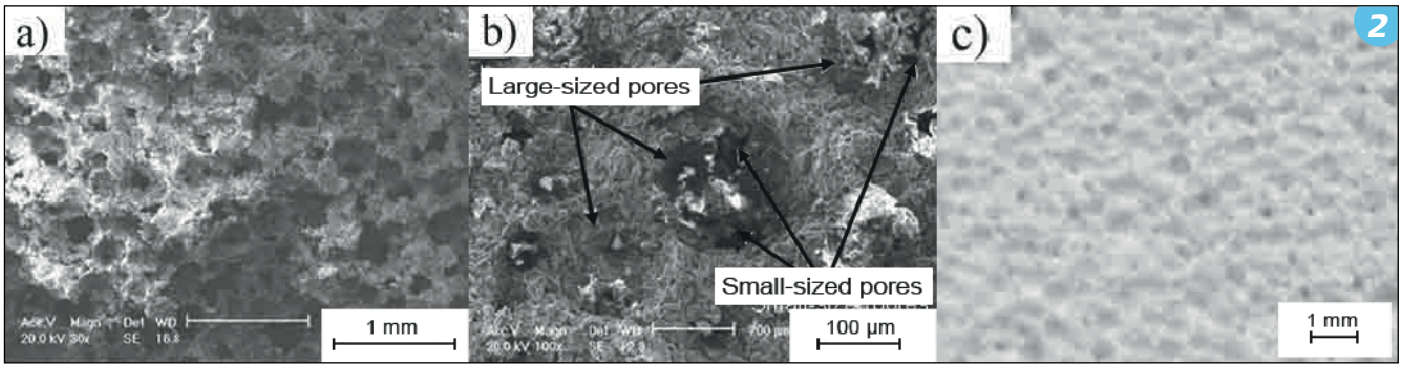

Fig. 2 • a) SEM images microstructure, b) a detailed pore microstructure and c) a cross-sectional view of mullite ceramics sintered at 1500 °C with a porosity of 80.25 vol.-% (solid loading 67.5 mass-%) [39]

29–60 % was in the range of 1.1–5.7 MPa. The properties of porous alumina ceramics can be well controlled by varying the alumina-to-yolk ratio and by tailoring the foaming process. Yin et al. [34] prepared porous Si_3N_4 ceramics with open porosity of 79.6–87.3 % and compressive strength of 2.5–22 MPa by using similar egg white protein as the foaming reagent. Gong et al. [39] prepared porous mullite ceramics with low thermal conductivity (as low as 0.09 W/mK) using starch both as consolidation and foaming reagent (heating at 75 °C for 1 h), which shows that porosity and compressive strength of the prepared porous mullite ceramics can be tailored by changing the solid loading of the slurry and the sintering temperature. The typical microstructure of the as-prepared porous mullite ceramics in Fig. 2 showed that the porous mullite ceramics were composed of a multi-modal microstructure, in which small pores are located in the internal walls of large spherical pores.

In a word, the main advantage of the temperature-induced gelling process is its non-toxicity and the main drawback of this method is that the solidification process of the foamed ceramic slurry should be carried out at a relatively high temperature (60–180 °C).

3 Sol-gel transition process

The sol-gel process has been used to stabilize foam during the direct foaming process and it has also been integrally used with emulsions or gas-liquid foam for the preparation of porous ceramics with a broad architecture diversity over the last decade [40–46]. Fujiu et al. [40] prepared porous silica ceramics using freon as the bubbling reagent by the sol-gel transition process. Destribats et al. [41] synthesized porous silica ceramics by mineralizing the continuous phase of oil-in-water pickering emulsions with the sol-gel process; the prepared porous silica ceramics had a porosity ranging between 73 % and 92 %, macro-cellular void diameters of 20–800 µm and a specific surface area (BET) of 700–900 $m^2 \cdot g^{-1}$. Alves-Rosa et al. [42] manufactured porous zirconia ceramics by a combined sol-gel and emulsification process and the obtained macroporous zirconia ceramics with high porosity (90 %) and low bulk density (0.40 g/cm^3) were prepared with 80 mass-% of decahydronaph-thalene. Beozzo et al. [43] investigated the effects of preparation parameters on the mechanical properties of porous zirconia ceramics using the sol-gel process associated with liquid foam templates. This showed that a high porosity (94 %) and a bimodal pore size distribution were obtained for the porous ceramic fabricated with 10 mass-% sodium dodecyl sulfate (SDS). The characteristics of the final porous zirconia ceramics are shown in Table 1. SEM results (Fig. 3) showed that macropores with an average size of about 30 µm were obtained in the final porous ceramics, and that the average size of the supermesopores increased with the increas-

Table 1 • Porous characteristics of porous zirconia ceramics prepared with 10 mass-% SDS and fired at various temperatures [43]

Thermal treatment	Total pore volume / $cm^3 \cdot g^{-1}$	Bulk density / $g \cdot cm^{-3}$	Porosity / %	Mean macropore size / µm	Mean supermesopores size / µm
500 °C	2.52 ± 0.01	0.358 ± 0.001	90.1 ± 0.3	10.9 ± 0.2	2.3 ± 0.2
600 °C	3.19 ± 0.05	0.293 ± 0.004	94 ± 1	13.6 ± 0.1	1.3 ± 0.1
800 °C	2.71 ± 0.05	0.331 ± 0.006	93 ± 2	14.0 ± 0.1	1.25 ± 0.07
1000 °C	2.6 ± 0.1	0.35 ± 0.02	94 ± 1	15.4 ± 0.1	1.15 ± 0.08

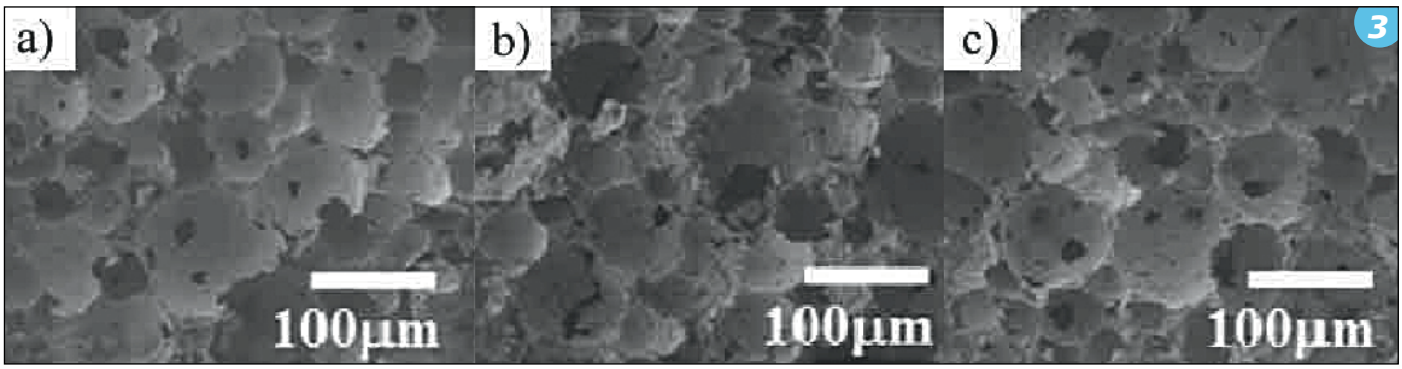

Fig. 3 • SEM images of porous zirconia ceramic prepared from liquid foams containing various amounts of SDS: a) 5 mass-%, b) 10 mass-%, c) 15 mass-% [43]

ing amount of SDS used; it reached a maximum value when 10 mass-% SDS was used in the process, and then decreased when using further increasing amounts of SDS.

Compared to the temperature-introduced gelling process, the sol-gel process not only offers a good possibility to tailor the microstructure of the prepared porous ceramics by controlling the synthesis parameters, but also greatly decreases the release amount of environmentally harmful substances during firing. However, it should be pointed that the sol-gel process is relatively expensive and complicated to compete with other current processes concerning the starting materials used. Moreover, the sol-gel process cannot be extended to prepare other kinds of porous ceramics except SiO_2 and Al_2O_3, due to a shortage of the corresponding metal alkoxide precursor.

4 Gelcasting process

In order to overcome the drawback that the foamed slurry should be coagulated at relatively high temperature by the temperature-induced gelling process and to prepare porous ceramics with a wide chemical composition, gelcasting processes using polymer as foaming and solidifying reagents instead of temperature-induced gelling and sol-gel transitions have been developed since the 1990s. Porous ceramics with controlled pore interconnectivity can be easily prepared without long heat treatments and complicated pyrolysis processes.

The in situ free radical polymerization of acrylamide monomers, originally developed for preparation of dense ceramics, was successfully used for setting foams and then preparing porous ceramics by Binner and Sepulveda [47, 48]. In this process, organic monomers are added to the foamed ceramic slurry and then in situ polymerized to form a cross-linked gelled structure that provides rigidity for the green body of porous ceramics. Sepulveda et al. [49–51] prepared porous alumina and porous hydroxyapatite ceramics using an organic monomer as the

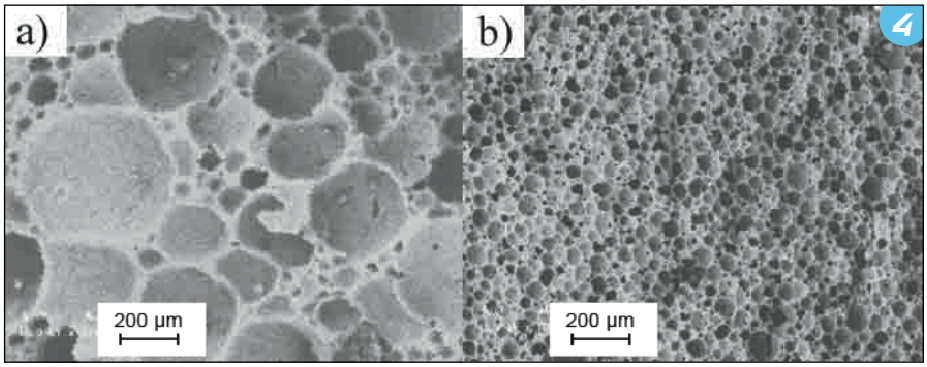

Fig. 4 • Microstructures of porous Si_3N_4 ceramics by adding a) PVA and b) CMC [57]

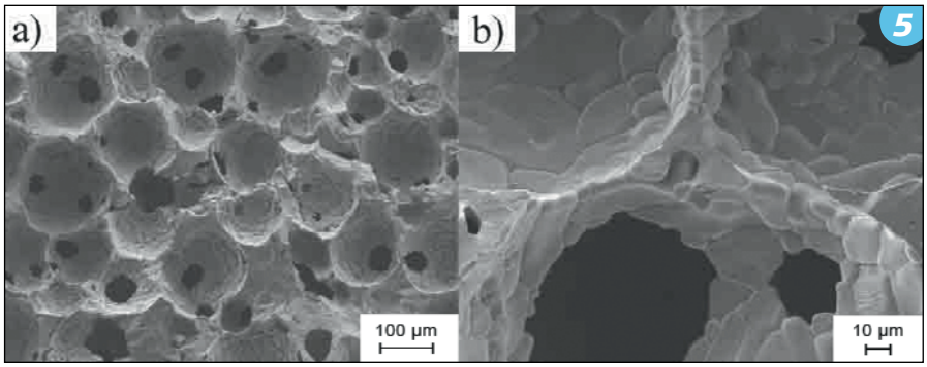

Fig. 5 • a) SEM image of porous ZrB_2 ceramics with 2 mass-% B_4C as the sintering aid, b) a detailed pore microstructure [58]

polymerizing reagent to stabilize foams in a N_2 filled chamber to insulate oxygen. The fabricated porous alumina bodies could be machined, and the final porous ceramic with a porosity ranging from 70 to 92 % and bending strength of 2–26 MPa consisted of a highly interconnected network with cell sizes of 30–600 µm. The compressive strength of the prepared porous hydroxyapatite (HA) ceramics with relative porosity ranging from 0.72 to 0.90 % and a pore diameter of 17–122 µm was in the range of 1.6–5.8 MPa. The permeability constants k_1 (Darcian) and k_2 (non-Darcian) of the prepared porous HA ceramics were strongly dependent on the porosity fraction and varied widely from 1.22×10^{-11} to 4.31×10^{-10} m^2 and from 1.75×10^{-6} to 8.06×10^{-5} m^2, respectively. Even though the porous ceramics prepared by this method usually possess a high strength due to the less flawed structure, it should be pointed that the gel-casting process suffers some drawbacks [52–54]. For example, this method cannot be used in the atmosphere because the polymerization process would be inhibited by the presence of oxygen; on the other hand, the acrylamide used as the main component of most of gelcasting systems is neurotoxic.

In order to overcome the above-mentioned drawbacks of the gelcasting process, many researchers have developed new gelcasting methods with oxygen insensitive and non-toxic cross-linking organic material as the polymerizing reagent. Mao et al. [55] prepared porous alumina ceramics using a water-soluble epoxy resin as the polymerizing reagents to solidify the foamed ceramic slurry in air. The permeability of the prepared porous ceramics with a relative density of

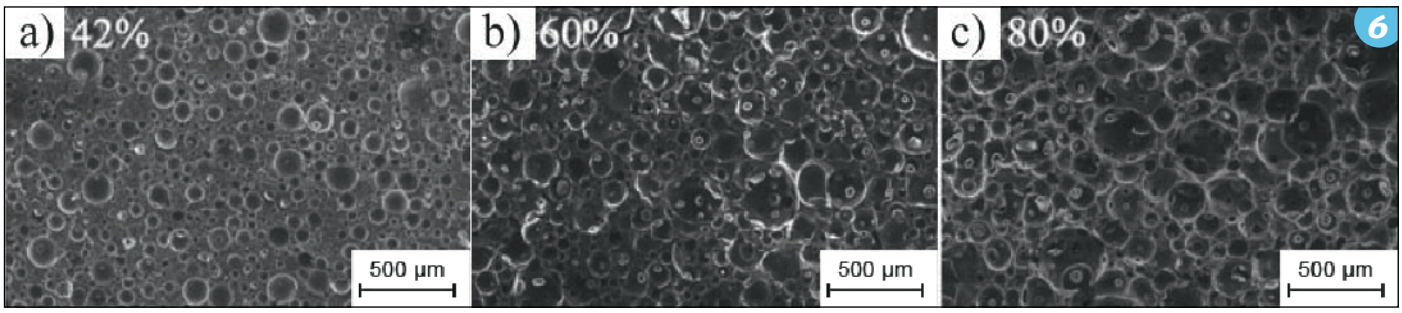

Fig. 6 • SEM images of porous alumina ceramics with different porosities using additions of a) 0.012 %, b) 0.02 %, c) 0.06 % Surf-E foaming reagent, respectively (porosity shown on the top of each picture) [59]

17 % and 36 % varied between 6×10^{-11} m^2 and 3×10^{-13} m^2. Kimet al. [56] prepared porous silica ceramics with a controllable pore size ranging from 70 to 150 µm and a porosity of 65–70 % by varying the sodium lauryl sulphate (SLS) amount in the three-phase foam slurry. Yu et al. [57] investigated the effect of carboxymethyl cellulose (CMC) amounts on the preparation of porous Si_3N_4 ceramics by a direct foaming method; no macropores or cracks were observed in the dried green body. Moreover, the sintered porous Si_3N_4 ceramics with a porosity of 60.6–82.1 %, an average pore size of about 16 µm and a flexural strength of 3.8–77.2 MPa had a uniform pore distribution. The microstructures of the porous Si_3N_4 ceramics prepared by using PVA and CMC as binders are shown in Fig.4. This indicates that the pore size of the porous Si_3N_4 ceramics prepared using CMC as binder is smaller than that of PVA, and on the other hand the pore distribution of the porous ceramics is also more uniform. Wu et al. [58] fabricated porous ZrB_2 ceramics by the direct foaming process; the effects of the foaming agent and the sintering aid on the flow behaviour of 45 vol.-% ZrB_2 slurries were also studied. Cellular ZrB_2 ceramics with the porosity of 54.9–72.6 % were obtained after gelcasting and pressureless sintering at 2100 °C, and a compressive strength of the prepared porous ZrB_2 ceramics with a porosity of 55 % was as high as 9.87±8.7 MPa. The microstructure of the prepared porous ZrB_2 ceramics sintered with 2 mass-% B_4C as sintering aids is shown in Fig. 5 which displays approximately spherical cells with a size of 60–200 µm.

Recently, to decrease the cost and the release of toxic volatiles during the sintering process of porous ceramics, Yang et al. [59] developed a novel gelcasting method for the fabrication of porous alumina ceramics using a water-soluble copolymer of isobutylene and maleic anhydride (commercial name: Isobam) as polymerizing reagent to solidify the foamed ceramic slurry, and the whole gelling process could be performed in air at room temperature. The prepared porous Al_2O_3 ceramics with a porosity of 20–89 % was one-step sintered at 1600 °C for 3 h; the microstructure of the prepared porous ceramics is shown in Fig. 6. This shows that the porosity and cell size increased with the foaming agent EMAL TD (Surf-E) content increase, and the average cell sizes of the prepared porous alumina ceramics were in the range of 60–220 µm. The compressive strength of the prepared porous Al_2O_3 ceramics with a porosity of 60 % was as high as 75 MPa. The advantages of this novel gelling system are that the only additive used can be easily removed before sintering; moreover, the process is environmentally friendly, since the addition of dispersant and polymerizing reagent is as low as 0.5 mass-%.

5 Summary and outlook

In summary, the direct foaming process is considered to be an effective method for the preparation of porous ceramics. The process can prepare both open and closed-cell structures with a wide range of cell sizes and porosities (up to 95 %). Foam stabilization is a critical step for the preparation of porous ceramics by the direct foaming process. Temperature-induced gelling of natural macromolecule compounds is a non-toxic processing route for the fabrication of porous ceramics, and the main drawback of the process is that the solidification process of foamed slurry should be carried out at a relatively high temperature. The sol-gel process can tailor the microstructure of the final porous ceramics and decrease the release of environmentally harmful substances. The disadvantage of the process is that it is hard to extend it to most kinds of ceramics, due to the shortage of the corresponding metal alkoxide. The gelcasting process is an easy and fast way to prepare required porous ceramics, and we think that the process will be widely used in the future because of the low amount of organic additive used and relatively low cost.

Acknowledgments

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