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# Processing of Ceramic Foams

Xiaojian Mao

**Abstract**

The ceramic foams have great importance in many industry fields. This chapter introduces the processing of ceramic foams by direct foaming technology. The structure of the precursor foams which attributes the final properties of the ceramic foams is described. Two different methods for foaming the ceramic slurry, as well as the techniques for foam consolidation, are presented in detail.

**Keywords:** ceramic foams, processing, foaming, stabilization, porosity

## 1. Introduction

Ceramic foams are tough foams made from ceramics, or ceramics with foam-like structure. It is a kind of porous ceramics with high porosity and sometimes called as cellular ceramics. Because of high amount of pores and surface, ceramic foams are especially suitable for filtering molten metals or hot gases, thermal protection systems, and heat exchangers [1].

The basic structural unit of the ceramic foams is composed of solid struts or walls and the empty cells surrounded by them. If the ceramic phase surrounds entire cells so that each cell is isolated from its adjacent ones, it is called as closed cell structure. If all cells are connected to each other with ceramic phase only in cell edges, it is called as open structure. In fact, ceramic foams often appear in a semi-open structure between the two ideal structures. The basic cell unit is the essential difference between the ceramic foams and general porous ceramics, which is actually a solid with isolated pores. And high porosity is its important characteristic. Gibson and Ashby [2] deem that there is a transition from ceramic foams to general porous ceramics with the relative density at about 0.3.

The earliest and still most common method for creating ceramic foams is the polymeric sponge replication method [3], with the products sometimes called as reticulated porous ceramics. In this
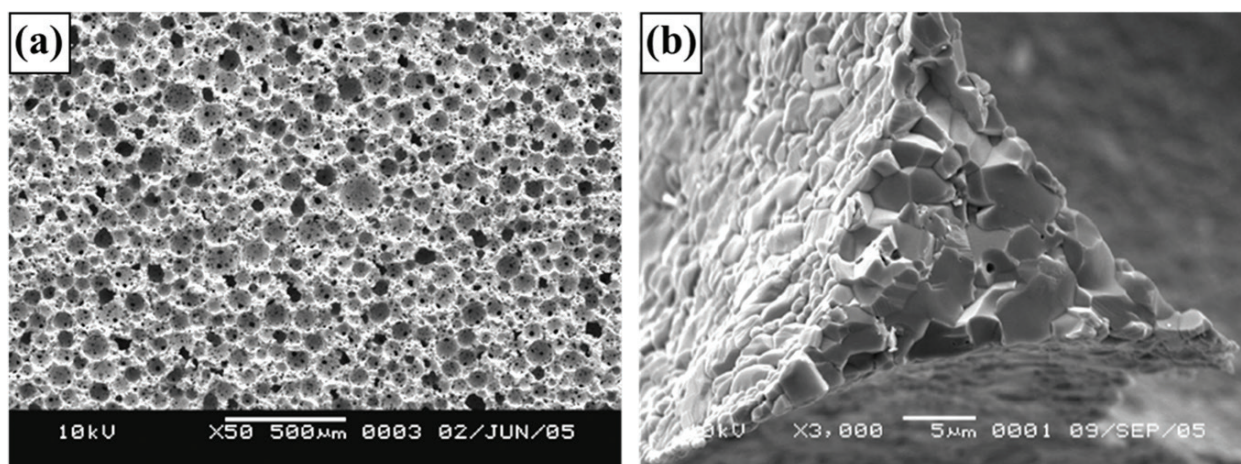
**Figure 1.** SEM photographs of ceramic foams consisted of (a) spherical cells and windows and (b) dense triangular struts.

method, a polymeric sponge with open pores is immersed into ceramic slurry, and after rolling to remove redundant slurry, the coated sponge is dried and pyrolyzed, leaving only the porous ceramic structure. Then, the resultant foam will be sintered for final densification to get required mechanical strength. This method is widely used because it is effective with most kinds of ceramic materials, such as silicon carbide, zirconia, silicon nitride, alumina, silica, mullite, and cordierite. However, large amount of gaseous by-product is released during pyrolysis, and consequently, leaving triangle hollows inside the ceramic struts. Cracking due to difference in thermal expansion coefficient is easy to occur [4]. Hence, there would be defects in ceramic foams fabricated by such polymeric foam replication technique, which led to lower mechanical strength [5].

Another technique to fabricate ceramic foams is direct foaming method. Ceramic foams are produced by incorporating air into a suspension or liquid media, which is subsequently set in order to keep the structure of air bubbles created. Then, the consolidated foams are afterwards sintered at high temperature to obtain high-strength foams [4]. This method can result in full dense struts without defects by polymeric sponge replication method. Hence, the mechanical strengths of the products are generally higher than those of reticulated porous ceramics. The characteristic of foams by this technique is that most cells are closed or semi-closed, depending on the air bubbles incorporated [6, 7]. **Figure 1(a)** shows the typical morphology of the ceramic foams prepared by the direct foaming method [8], and **Figure 1(b)** is a cross-sectional photograph of the dense struts. This chapter describes the processing of ceramic foams by direct foaming method.

## 2. Liquid precursor foams

### 2.1. Structure of foams

A bulk foam is a substance formed by trapping gas air bubbles in liquid or solid. A bath sponge and the top of fresh beer are examples of foams. In most cases, the volume of gas is

large, with thin films of liquid or solid separating the regions of gas. The equilibrium structure of foam is an elegant and well-defined arrangement of films, plateau borders, and junctions. The bubbles which are pressed together to form the foam are separated by thin films. The films meet along a line or curve, forming a liquid-filled interstitial channel called a plateau border. Where several plateau borders meet to form an interconnected network, they do so at a junction [9].

## 2.2. Liquid foams

Liquid foams are thermodynamically unstable systems due to their high gas-liquid interfacial area. Several physical processes take place in wet foams to decrease the system free energy, leading to foam destabilization. The main destabilization mechanisms are drainage (creaming) and coarsening (Ostwald ripening). Drainage is the physical separation between the gaseous and liquid phases of the foam because of the effect of gravity. In draining foams, light gas bubbles move upwards, forming a denser foam layer on the top, while the heavier liquid phase is concentrated on the bottom, as illustrated in **Figure 2** [4]. Coarsening is the gradual change of the foam structure due to gas diffusion through the films. This diffusion is driven by the pressure differences between bubbles. Small bubbles have high pressure, so they lose gas and disappear. Thus, the average bubble size increases with time.
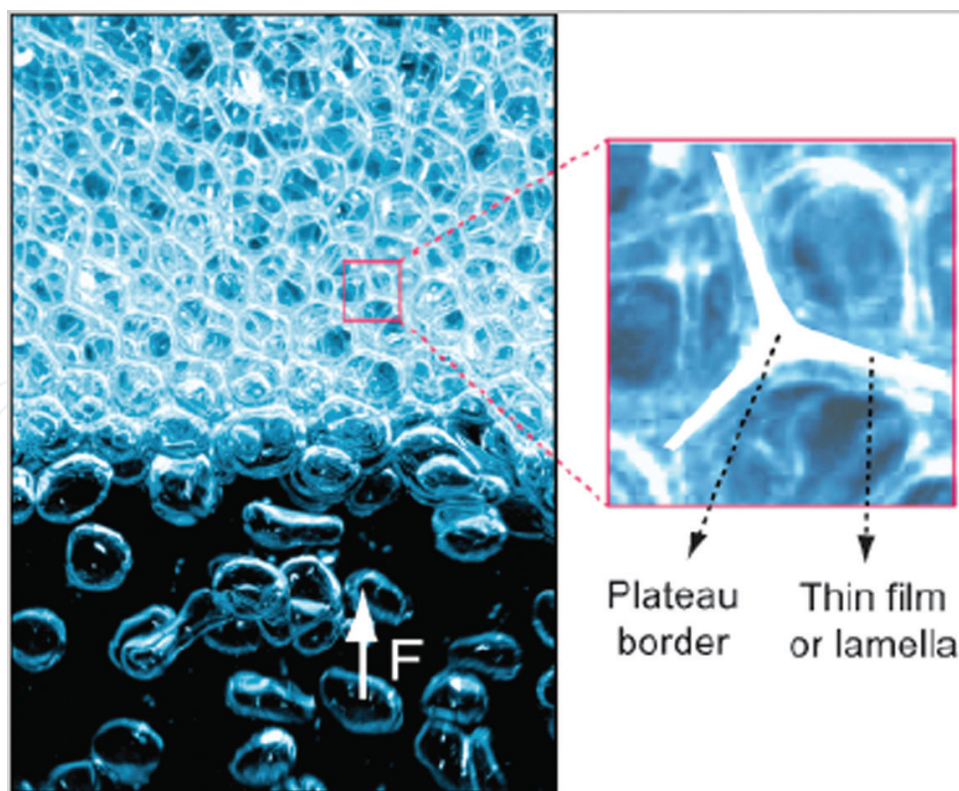


**Figure 2.** Photograph of foam drainage and foam structure [4].

Generally, real liquid foams are only stable if they contain surfactants. Good foams usually contain complex molecules that toughen the walls of the bubbles. Milk fat, for instance, serves this purpose in whipped cream. The way the bubbles stick together or slip past one another determines how the foam behaves.

### 2.3. Particle-stabilized wet foams

Solid particles with tailored surface chemistry have lately been shown to efficiently stabilize gas bubbles upon adsorption at the air-water interface. The attachment of particles at gas-liquid interfaces occurs when particles are not completely wet in the liquid phase or, in other words, are partially lyophobic (hydrophobic if the liquid is water). The position of the particles at the interface is ultimately determined by a balance between the gas-liquid, gas-solid, and solid-liquid interfacial tensions, as shown in **Figure 3** [10]. A simple way to describe the particle position at the interface is through the contact angle formed at equilibrium through the liquid phase. Slightly lyophobic particles remain predominantly in the liquid phase and exhibit a contact angle <90°, leading to the formation of air in water mixture, that is, foams [4].

In this method, the amphiphiles added to the suspension let the particles partially hydrophobic by adsorbing with its polar anchoring group on the surface and leaving a short hydrophobic tail in contact with the aqueous phase. Studart et al. [4] summarized some amphiphilic compounds, such as valeric acid, propyl gallate, butyl gallate, and hexyl amine, for surface modification for different particles. After surface modification, air can be easily incorporated by mechanical whipping, injection of gas stream, or initiation of a chemical reaction that releases gaseous by-products directly into the initially fluid suspension.

However, the particle-stabilized wet foams are not strong enough to resist the stress during drying. Hence, they still need to be strengthened before water evaporation, either by coagulating the particles within the foam films or by chemically gelling the foam liquid phase [11].
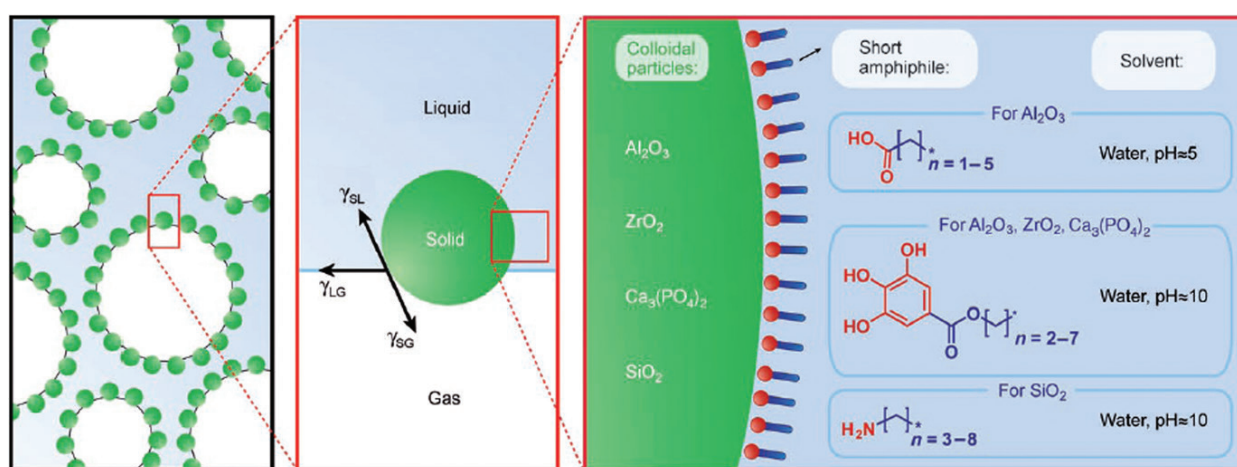


**Figure 3.** Diagrammatic sketch of the particle-stabilized foams [10].

## 2.4. Foams of ceramic slurry

In order to manufacture ceramic foams, the liquid foams have to contain abundant ceramic particles in the liquid phase, which are going to be sintered as the main component of the corresponding ceramic foams. **Figure 4** shows the diagram of the related structures for precursor foams and the resulting ceramic foams. The bubbles, which occupy the most volume of liquid foams, turn into cells of the ceramic foams. The films, which comprise liquid and ceramic particles, transform into the cell walls. Generally, the central part of the films is too thin to keep intact during sintering. Hence, there are commonly windows in the cell walls between two neighbor cells. This kind of common constitution is called as semi-open structure. If the films are extremely thin or the solid contents are too low, only plateau border is survived after sintering corresponds to the struts between three or more cells. That generates the strict open pore structure.

Generally, the solid content influences the rheology of ceramic suspension. High solid content contributes to high viscosity and shear thin behavior. And the solid content would also associate with the final structure of the ceramic foams. Consequently, it is a practical way to adjust the porosity and structure of ceramic foams by controlling the solid content of the original suspension. Mao et al. [12] manufactured alumina foams with different morphology by changing the solid content using direct foaming and gelcasting method. **Figure 5** shows the rheological flow curves of suspensions with different solid contents. All suspensions reveal pseudoplastic behavior, and the viscosity increases with solid content at the measured shear rate range. In the fabrication of ceramic foams, a slight pseudoplasticity could favor the generation of foams since lower viscosities are obtained under high speed whipping, and the foam stability would be improved because the viscosity recovery under static condition delays the collapse of fluid films around the bubbles.

The relative density of final alumina foams increases with the solid loading, while other processing conditions are constant, as indicated in **Figure 6**. The reason is that high solid content results in high viscosity, corresponding to low foaming capacity and high relative density. It
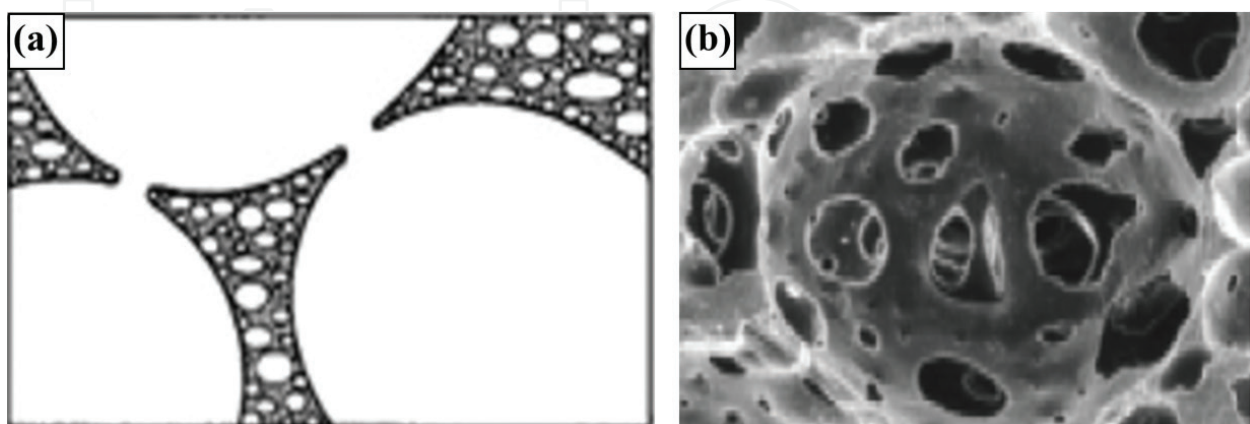


**Figure 4.** Structural diagram of (a) precursor foams and (b) the sintered ceramic foams.
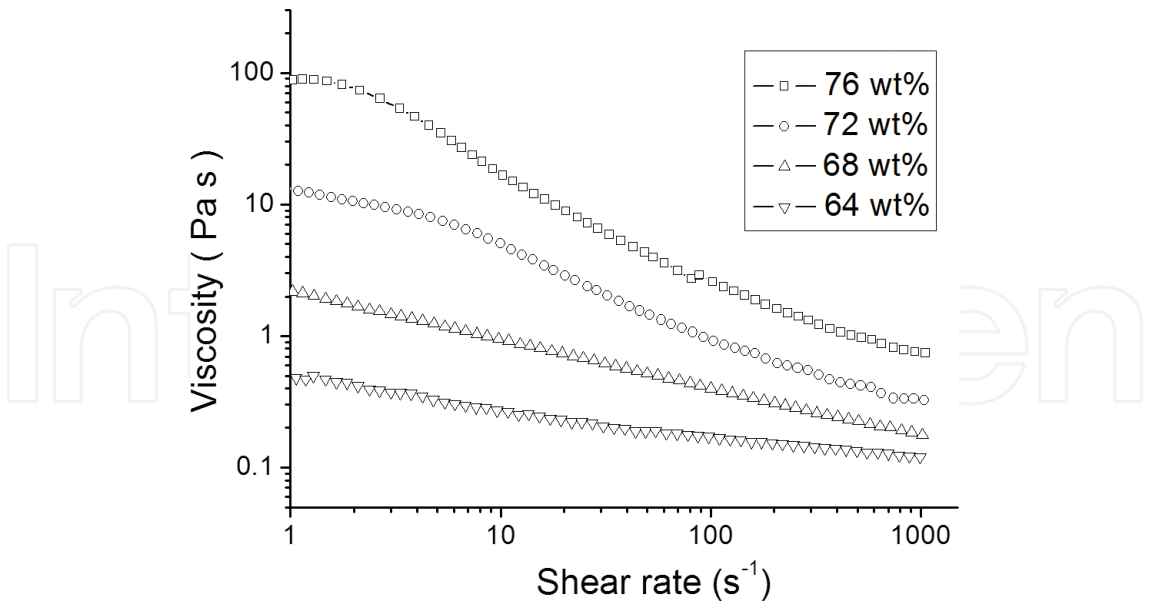
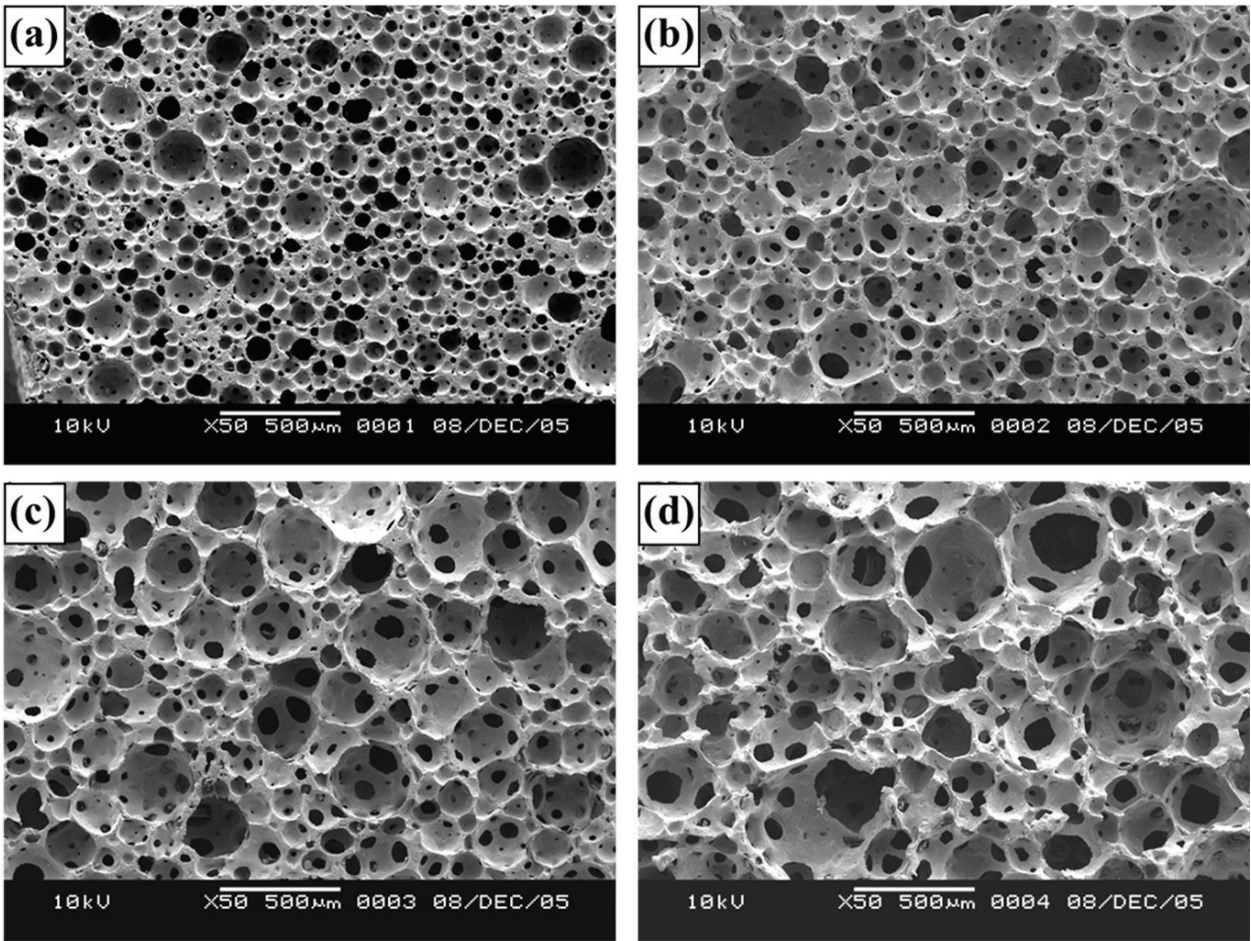**Figure 5.** Rheological flow curves of suspensions with different solid contents [12].



**Figure 6.** SEM micrographs of alumina foams with suspension solid content of (a) 76 wt%, (b) 72 wt%, (c) 68 wt%, and (d) 60 wt% [12].

can also be seen from **Figure 6** that with the decrease of the relative density, both the mean cell size and the window size are increased.

In order to evaluate the influence of the size of ceramic particles, Mao et al. [13] introduced granule particles into the original powders to manufacture the alumina foams. The coarse powder was manufactured by grinding the presintered foams obtained by the fine powder, in order to keep similar sintering ability to fine powder. The flexural strength of the sintered foams with the coarse powder is 25% lower than that by original powder. And the permeability of foams using the coarse powder is about 30% higher than that by original powder. The drop of flexural strength and the rise of permeability are related to high degree of open pores.

# 3. Foaming techniques

Foams and foaming phenomena are common and important in our daily lives. While putting some shaving cream or soap on our faces, and rub gradually, we will create a truly bizarre substance, which are most gas and little bit of liquid. When we whisk air into egg white or cream, bubbles form and linger because the proteins present in these viscous liquids stretch around bubbles and trap them. The foams spout out from the compressed bottle, when we style our hair with mousse.

All these techniques would be applied in the manufacture of ceramic foams. The foaming of ceramic slurries involves dispersing gas in the form of bubbles into ceramic suspension. There are two basic approaches for achieving this: (1) incorporating an external gas by mechanical frothing, or injection of a gas stream and (2) evolution of a gas in situ [14]. In order to stabilize the bubbles developed within the slurry, the surface tension of the gas-liquid interface need to be reduced by, in most cases, adding surfactant or by sometimes partially hydrophobic particles. In some cases, water-soluble polymers are added into the slurry to modify the viscosity, which will affect the foaming results and the stability.

## 3.1. Incorporation of an external gas phase

One of the ways foam is created is through dispersion, where a large amount of gas is mixed with a liquid. Mechanical stirring is the most common technique for gas dispersion. Electric beater or household whisk is convenient choice for foaming of ceramic slurries [15]. The whisking procedure involves incorporating with air-forming bubbles, and at the same time, the bubbles flow up and break because of drainage and coalescence. Hence, generally, surfactant is necessary to reduce the surface tension to stabilize the bubbles. When the speed of bubble generation and burst become equilibrium, the maximum volume of the foam is obtained.

**Figure 7** shows the foam volume versus stirring time for alumina suspensions containing two different foaming agents, Triton X114 and Tween 80 [16]. The foam volume increases gradually up to a maximum after approximately 4 min of agitation. During this initial stirring period, gas is entrained into the suspensions and liquid is drawn around each bubble until a thin film is formed. Subsequently, the surfactant molecules of the foaming agent transfer from
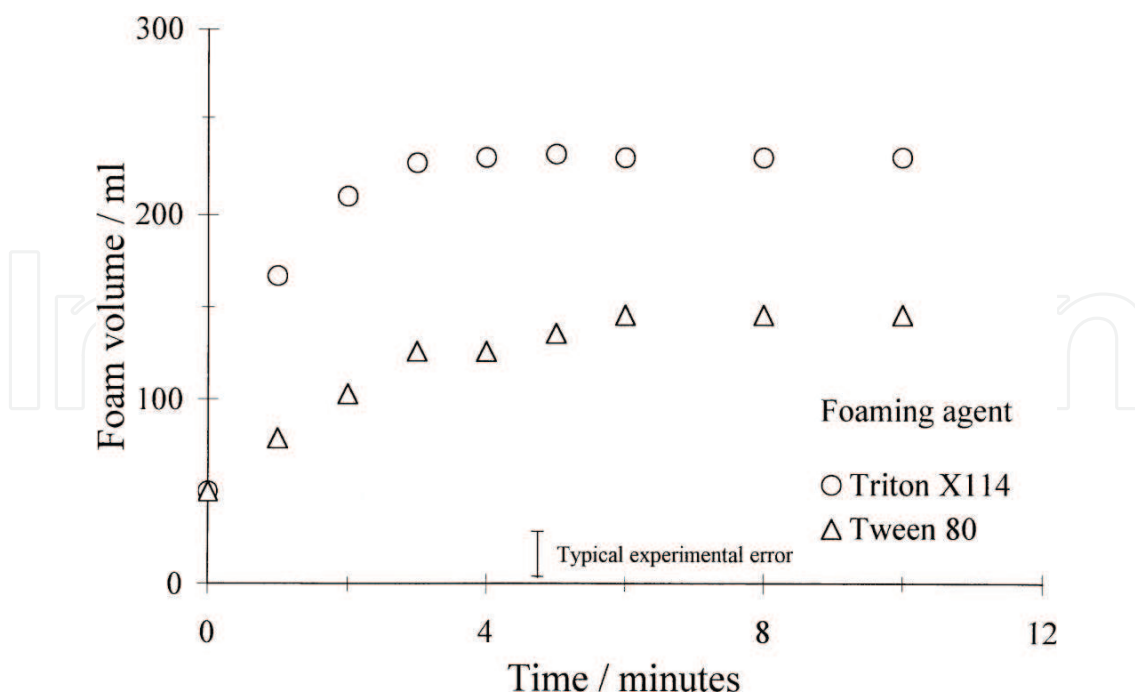
**Figure 7.** Foam volume generated with two different foaming agents [16].

interior of the suspension toward the newly created surface, decreasing the surface tension. Increasing the surfactant concentration accelerates this transfer and hence increases the foaming capacity [17]. Sepulveda considers that the maximum foam volume is associated with a minimum thickness of film that can sustain a stable foam. When most surfactant molecules have attached themselves to the gas-liquid interface, the stabilization of new films is no longer possible and the volume increase becomes negligible.

### 3.2. In situ gas evolution

The foaming technology of this theme is the presence of a foaming agent that decomposes due to heat or a chemical reaction to generate a gas within a ceramic suspension. Kim [18, 19] used the mixture of the cross-linked polycarbosilane and polysiloxane as the preceramic polymers to manufacture the SiOC foams, which were pressed into disks and CIPed at 340 MPa. The green compacts were placed in a pressure chamber to saturate with gaseous $CO_2$ under a pressure of 5.5 MPa. Then, a thermal dynamic instability was introduced by rapidly dropping the pressure at a rate of 2.9 MPa/s. The foamed preceramic specimens were further cross-linked, then pyrolyzed, and sintered at 1200°C in nitrogen. Takahashi et al. [20] used the blend of methylsilicone resin and polyurethane precursor to prepare the SiOC foams. The foamed blend was prepared in two steps. The first step was the addition of methylsilicone resin dissolved in $CH_2Cl_2$ to the mixture of the polyols, the amine catalysts, the surfactant, and the additional dichloromethane. The second step was the addition of polyisocyanate to the solution obtained in the first step. The expansion started during mechanical stirring by the evaporation of the solvent caused by the exothermal reactions occurring in the solution.

## 4. Foam consolidation

In direct foaming method, ceramic foams are prepared by introducing large amounts of air bubbles into the slurry. The foam is essentially a metastable system, with some bubbles shrinking and others gathering. It is important to consolidate the foams in certain period, to keep the cellular structure during further heating procedure [21]. In order to prevent the foams from drainage or coarsening, it is necessary to accelerate the consolidation speed and obtain a higher strength. Suitable consolidation method would bring uniform and dense struts, which is a benefit for mechanical properties of the resulting ceramic foams.

### 4.1. Freezing

Freezing method is one of the practical methods to consolidate the foamed slurry [22]. Verma et al. [23] manufactured silica foams with 85 vol% porosity content from ceramic slurries containing ovalbumin as binder along with additives of sucrose and colloidal silica by combination of direct foaming and freeze-casting routes. The foamed slurries were poured into vaseline-coated aluminum molds and cooled using liquid nitrogen for instant freezing of porous structure. The frozen samples were freeze-dried at a low temperature for 24 h. After drying, the dried foam was heated to 1150°C to remove the binder and sinter the pore walls. The advantage of the freezing method is that extra consolidation agent is unnecessary. However, during freezing procedure, the liquid solvent, for example, water, will transfer to solid crystals which entrapped between the agglomerated ceramic particles at the films. These crystals will leave micropores after the evaporation of solvent. Rapid freezing of the solvent leads to formation of fine ice crystals, while long-time freezing procedure would enlarge the size of the crystals. The corresponding large pores may not be removed during sintering and, hence, lead to lower mechanical strength. The freezing time has to be prolonged to cool down the temperature of inside parts for large-sized bulk foams since the foamed slurry is a thermal insulator, which indicates that the frozen crystals will grow during consolidation of large-scale products. Thus, the freezing technique might not be good for high-strength foam production.

### 4.2. Natural polymer denaturation

Some natural polymers from animal and plant sources have the properties of liquid-solid transition due to denaturation which has potential applications in the consolidation of foamed slurries. Protein and polysaccharide including starch, agar, and cellulose are often used to manufacture ceramic foams and porous ceramics [23–25]. Proteins are high molecular compounds, which are formally understood as condensation products of amino acids. The amphiphilic character of these molecules causes a decrease of surface tension, therefore good foaming properties. These foaming properties are influenced by the amino acid sequence or rather the number of polar and apolar side chains as well as molecule flexibility [26]. After foaming, the foamed slurries are consolidated by changing conditions, for example, adding acid or heating over 60°C, which would trigger the irreversible changes in the spatial structure of the protein molecule.

Garrn et al. [26] used albumin, a major constituent of blood, as a model binder to an aqueous powder suspension to produce ceramic foams. Foaming was done in a planetary mill using PE-milling pots for 15 min. A fine cellular foam structure with approximate diameter of 50–300 μm was formed. Thermal consolidation was done in a conventional household microwave oven with a maximum microwave power of 900 W. After burn out and sintering, final densities in the range from 8 to 20% were achieved. Fish collagen and egg white [27, 28] are other specific examples of protein applied for ceramic foams. They were added into ceramic slurries which would be stirred to become foams. The foamed slurries were then heated at 80°C or higher for consolidation, attributing to the gelation of protein.

Methylcellulose and polysaccharide, which have similar transformation as protein in case of heating, are also typical agents for consolidation of foamed ceramic slurries. Mao et al. [17] manufactured silica foams based on the generation of foams from composite slurries with cassava starch. These slurries combined with surfactant were vigorously whisked for about 5 min to make foam structure. The as-foamed slurries were then preheated in a microwave oven with a power of 400 W for 60 s, followed by setting in a 70°C oven for 30 min to consolidate the foam structure. After sintering, the resulting silica foams with the relative density of 18–30% were obtained. Because the cassava starch is not soluble in water, the particles will residual pores inside cell walls and struts after debindering and sintering. Hence, the direct foaming and starch consolidation method can produce porous ceramics with hierarchical structures, as shown in **Figure 8**. **Figure 8(a)** shows clearly the spherical cells with the average size of about 50 μm, while **Figure 8(b)** reveals the pores averaging about 10 μm in the cell walls. **Figure 8(c)** further indicates small voids inside ceramic matrix. It can be inferred that the large-sized cells, moderate-sized pores, and small-sized voids were originated from bubbles, elimination of starch particles, and interstices among the silica grains, respectively.

These natural polymers which can be operated in laboratory environment, for example, simply heated at 50–80°C, are excellent agents for ceramic foams. They are environmentally friendly and low cost and, hence, are widely used. However, the consolidation procedure needs heating of the foamed slurries, which would lead to the expansion of air bubbles. The metastable structure would change during the temperature change, which should remain some defects in final ceramic foams.
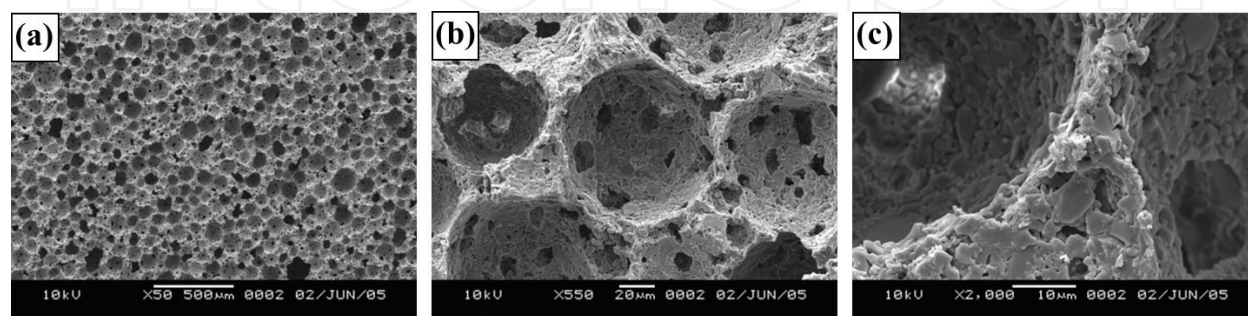


**Figure 8.** SEM micrographs of sintered ceramics with details of (a) large-sized cells, (b) moderate-sized pores in cell wall, and (c) small-sized voids among silica grains [17].

### 4.3. Gelcasting

In recent years, the gelcasting method was developed to manufacture ceramic foams by solving the shortcomings of the mentioned natural polymer substances which need heating for denaturation. The water-soluble small molecule compounds are added into the slurries, which will form a gel through radical polymerization. The method was first proposed by Smith [29], which combines the foaming and gelcasting processes, resulting in wet foams with high strength for drying and further handling. Sepulveda and Binner have done a lot of work on the gelcasting of foams, which has been shown to be useful in a variety of ceramic systems such as zirconia, alumina, and hydroxyapatite [30]. The benefit compared with using polymers is the ability to formulate slurries with a lower viscosity, because the size of the organic molecule is smaller, so that higher solid contents can be achieved with good packing densities and excellent green strengths [31]. Such a process yields cellular structures with porosity varying from 40% to >90%, with pores closed or open depending on pore fraction. Mechanical strength of sintered foams is higher than that obtained by other routes, because of the spherical pore shape associated with fully dense matrix [15].

**Figure 9** shows the flow chart for production of ceramic foams by gelcasting of foams. Ceramic slurries with monomers and surfactants are vigorously whisked under inert gas atmosphere to form foams. Afterwards, catalyst and initiator are added to trigger the polymerization reaction, forming a strong three-dimensional gel net. The concentration of these reagents is designed to produce an induction period such that polymerization will be initiated immediately after casting. Within the time allowed by the induction period, the foams could be placed in a desiccator with the pressure reduced using a vacuum pump to produce foams with cell size larger than those obtained directly through foaming [16]. The excellent green body strength is the main advantage of the gelcasting of ceramic foams, which may maintain a porous structure with the porosity up to 90%, compared with other consolidation methods. The investigation to the sintered foams confirms that the solid matrix has very high density, which is much perfect than those by polymeric sponge replication method. Sepulveda produced alumina foams with the bending strength in the range of 2–26 MPa, while their relative density varied in 8–30% [30].
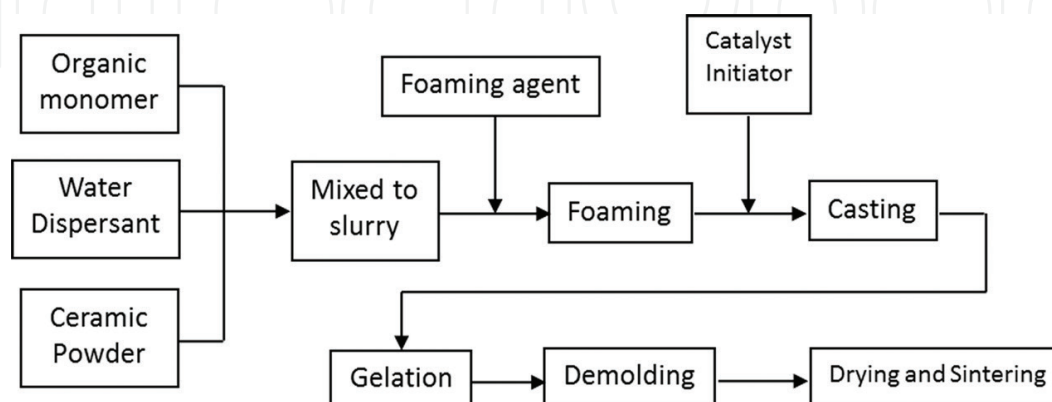


**Figure 9.** Flow chart for production of ceramic foams by gelcasting of foams.

However, the usual monomers are acrylamide derivatives, and the polymerization is a free radical reaction which is inhibited by oxygen. For example, just 0.2% oxygen was sufficient to inhibit the reaction completely in foamed suspensions [30]. Thus, the foaming and polymerization procedures have to be carried out in a $N_2$-filled chamber to insulate oxygen.

Mao et al. [32] developed a novel gelcasting system based on epoxy resin and polyamine hardener, which could be operated in air atmosphere, because the polymerization between the epoxide group of the epoxy resin and active hydrogen of amine is a nucleophilic addition reaction which is not affected by oxygen in atmosphere. This gelcasting system was then applied to manufacture ceramic foams with some modification [12]. Aqueous suspensions with solids loading of 60–76 wt% were prepared by mixing alumina powder, dispersant, and 5 wt% polyethyleneimine solution. Vigorous stirring about 5 min was applied after adding the surfactant to generate foams. For setting the fluid foams, 10 wt% sorbitol polyglycidyl ether based on the premix solution was added with further stirring about 30 s. The foamed suspensions were immediately poured into plastic molds and sealed at room temperature for gelation.

Yang et al. reported a novel single-component water-soluble copolymer of isobutylene and maleic anhydride, with a commercial name of Isobam, which could be used as both surfactant and gelling agent with the addition much lower than normal gelation systems [33]. Yang et al. developed this system for the consolidation of ceramic foams. A small addition of 0.3 wt% Isobam based on alumina powder is sufficient to consolidate liquid foams and maintain the wet foams for further treatments [34]. Small additive amount is benefit for further heat treatment because the exhaust gaseous by-product can be dramatically reduced. It was confirmed that Isobam could be applied to manufacture variety of ceramic materials, such as mullite and $Yb_3Al_5O_{12}$ [35].

### 4.4. Sol-gel

Sol-gel method has been widely used in the preparation of powder, film, and bulk materials. Since the processing of sol-gel is actually a liquid-solid transformation, it can be used to consolidate the liquid foams without any other additive. The advantage of this route is that no contamination is involved, which is suitable for producing high-purity ceramic foams. Silica foams and silica-contained ceramic foams have been manufactured [36–38]. Commercial $SiO_2$ sol or the hydrolyzate of the precursor tetraethoxysilane was modified by adding acid to the pH value in the range of 5–6. After adding surfactant, the sol is incorporated with air by mechanical stirring or in situ gas evolution. Then, the foamed sol will be gradually consolidated with the sol transfer to gel. The porosity and the pore size distribution may be controlled by changing the viscosity and foaming technology. The silica-based sol-gel system has been used in many ceramic foams, such as silica, boehmite, and zirconia [21]. Pereira et al. [37] manufactured bioactive glass and hybrid scaffolds for bone tissue engineering by sol-gel method. TEOS and calcium chloride were used as the silica and calcium precursors, respectively. The starting sol was prepared by hydrolysis of TEOS in the presence of 1 N hydrochloric acid solution with subsequent addition of calcium chloride. PVA solution, Teepol surfactant, and 5 vol% hydrofluoric acid solution were added to a 40-ml aliquot of the

sol, and the mixture was foamed by vigorous agitation. HF was added in order to catalyze the gelation. The foamed gels were cast, aged at 40°C for 72 h, and dried at 40°C for 120 h. Final glass and hybrid foams can be obtained with a high porosity varying from 60 to 95% and macropore diameters ranging from 10 to 600 μm.

## 5. Drying and sintering

The consolidated wet foams are a mixture of gas, liquid, and solid, which need to be dried and debindered before sintering to final ceramic foams. Since there are large amount of bubbles dispersed in the bodies, the green strength is much lower than that of normal ceramics. Hence, both drying and debindering procedures should be carried out carefully. However, the bubbles, especially the connected bubbles, would become channels for water, solvent, or pyrolyzate to escape. Generally, the drying and calcination speed should be slowed down to avoid possible crack.

The foamed green bodies need to be sintered to get sufficient strength for further applications. It is important to modify the sintering schedules to get dense and strong struts and cell walls, to increase the mechanical properties of the ceramic foams. The sintering for ceramic foams is not get equivalent research intension as for foaming and consolidation, since the sintering behavior is dominantly decided by the powders. Especially for the particles inside the struts and the cell walls, the coordination particles are same within normal ceramics. However, for those particle located on the surface of cell walls or in the tip of the strut edges, their coordination particles are less than in dense green bodies. **Figure 10** shows the SEM microstructures of the fracture surface of the struts and the edge of the cell window. We can see clearly that grain size inside the struts is larger than that near the cell wall surface. And the gran size becomes much smaller when the location shifts to the tip of the triangle. The ceramic sintering theory seems not simply suitable to describe the ceramic foams. The difference for grain size
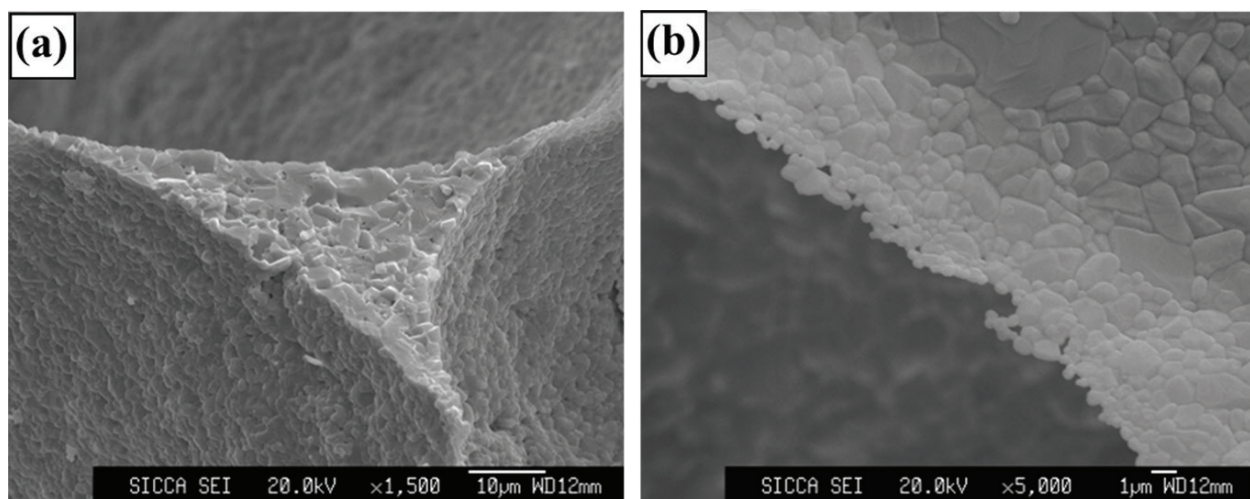


**Figure 10.** Microstructure of alumina foams: (a) fracture surface of the struts and (b) edge of the cell window.

is related to the particle coordination, where large coordination number corresponds to large grain size. The possible reason is that more coordination particles indicate abundant mass resource for grain growth.

## 6. Summary

Due to its current and potential great application, ceramic foams attracted distinct attentions in past decades with new process routs constantly being developed and reported in the scientific literature and at conferences. As a kind of porous ceramic with special structure, the ceramic foams gradually play irreplaceable roles in many industry fields, such as diesel particulate filters, interpenetrating composites, high-temperature thermal insulators, and biomedical applications. It is very important and valuable to explore novel manufacture routes and continuously improve the performance of ceramic foams. Whereas the polymer replication process is advanced to be in commercial use for decades, now the slurry foaming techniques are developed rapidly, which yields ceramic foams with different morphologies, and hence different properties and potential applications. This provides much greater choice for the end user and far greater potential for the tailoring of structures to meet specific end-sue requirement.

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