



Research Trend in the Processing and Application of Macroporous Ceramic Components

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Abstract - Porous ceramic components with decently controlled porosity offers remarkable advantages in industrial and structural applications such as fluid filtration, thermal insulation and scaffolds for bone tissue engineering. In this review study of porous ceramic components, requisite processing techniques necessary for the development of porous ceramics with imbued microstructural model intended for a specific application. An appraisal of the fabrication was made with respect to their economic viability wherein cost effective methods having great potentials in decently controlling the pore network imbued within the host ceramic matrix was preferred over the capital intensive counterparts.

Keywords: Processing routes, Sacrificial fugitives, Replica template, Porous ceramic scaffolds

Introduction

With the vast groundbreaking technological progression in the field of material science, porous ceramic materials have high expectations to function decently in a multiplicity of industrial applications ranging from filtration, thermal insulation systems, absorption, catalyst and catalyst supports to light weight structural components (Ohji & Fukushima, 2012; Kumar & Kim, 2010; Li & Li, 2012; Ren et al., 2013; Roohani-Esfahani et al., 2013; Roy et al., 2011). The utilization of porous ceramic materials for industrial and structural applications has received a tremendous acceptance amidst various end users owing to their high thermal stability, chemical resistance, good wear resistance, poor conductivity, high hardness etc. This group of materials has surged the interest of researchers by delving further into advancing the development of ceramic products that can suit other specific requirements. Thus far, studies have shown one of the major setback in broadening the application range of porous ceramics to be the choice of fabrication route for developing porous ceramic components. Utilizing the appropriate processing route promotes the systemic control of the pore network within the product's microstructure which in turn expands their existing application range (Hammel et al., 2014). For instance, the processing of scaffolds with imbued pore network for bone tissue engineering application requires establishing a tradeoff

between the requisite mechanical properties of the scaffolds as well as the imbued porosity level (Zamanian et al., 2013; Ghazanfari & Zamanian, 2013; Tripathi & Basu, 2012; Roohani-Esfahani et al., 2013). To a large extent, ample homogenous porous ceramics have been commercially produced via the exploitation of state-of-the-art processing technologies. Finally, the processing techniques are comprehensively discussed with regards to their economic viability and the emerging microstructural features.

Processing Routes of Porous Ceramics

In spite of the wide-ranging application of porous ceramics, consideration of the processing technique is highly recommended as the porosity level, pore morphology and the resultant mechanical strength are functions of these processing routes. More so, it is noteworthy that the application areas determines to a large extent the choice of processing method to be employed in the fabrication of porous ceramic materials. There are various processing routes available for developing porous ceramics with decently tailored pores. As shown in Figure 1, the processing routes can be categorized under the following; (i) partial sintering, (ii) replica template, (iii) sacrificial fugitives and (iv) direct foaming.

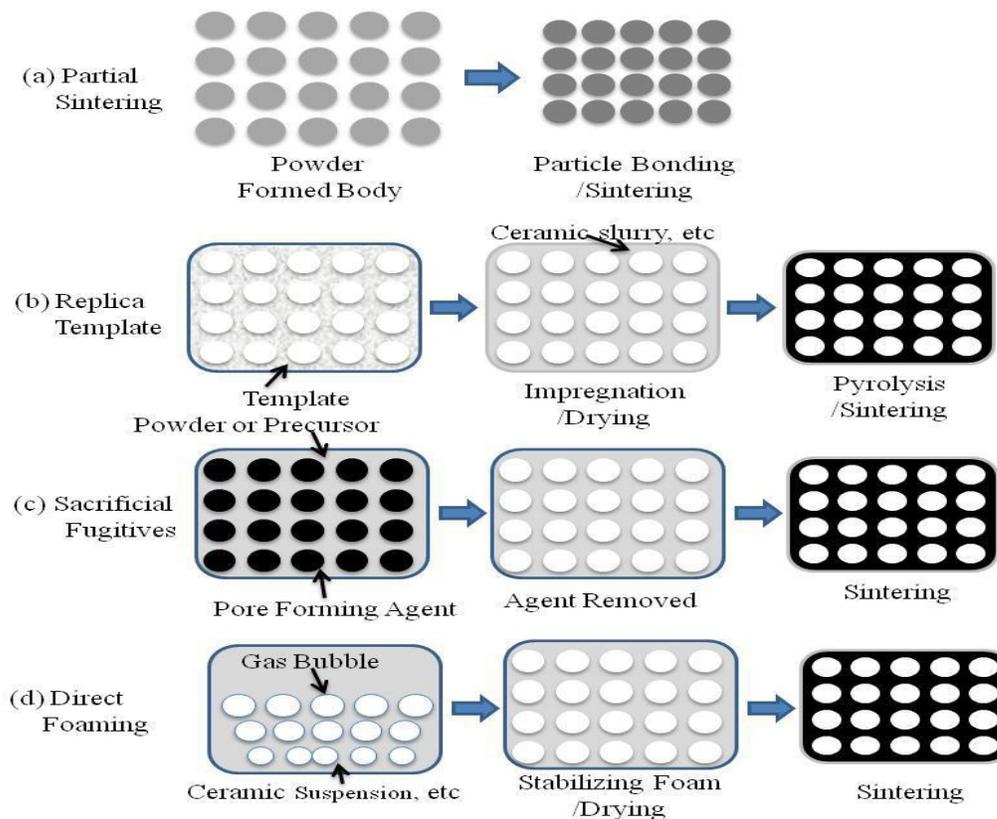


Figure 1: Representative processing techniques for macro-porous ceramic components (Ohji & Fukushima, 2012).

Partial Sintering Method

According to Yang et al., one of the easiest methods for preparing porous ceramics with homogeneously distributed pores is the partial sintering technique (Yang et al., 2009). This process can be utilized in the sintering process of green compacts or sintering of raw powder mixture wherein solid state reactions occur and a network of pores emanates within the host matrix. Furthermore, the research survey of Ohji &

Fukushima revealed the yardstick for the formation of the desired pore size in partially sintered porous ceramics to be the size of the raw powder which must be geometrically in the range of two to five times larger than that of pore (Ohji & Fukushima, 2012). More so, the authors gave three parameters for porosity reduction which are; rise in operating pressure, sintering temperature and duration of heat treatment.

In their investigation, Tuyen et al. reported the fabrication of reaction-bonded silica nitride (RBSN) bodies through the partial heat treatment of the RBSN bodies at temperature of 1550°C and 6wt% Y_2O_3 -2wt% MgO as additives (Tuyen et al., 2009). Their findings indicated that the phase ratio, porosity and mechanical strength of the porous RBSN bodies subjected to the partial sintering process at 1550°C for 9h were 99%, 43.2% and 141MPa respectively. As compared to higher sintering temperature at 1850°C for 6h, the mechanical strength and porosity level of the porous RBSN bodies were about 285MPa and 38% respectively. It is worthy of note that the porosity of the partially sintered bodies were higher than those sintered at 1850°C. However, the compression strength of the partially sintered bodies was remarkably lower as compared to those sintered at higher temperature.

Replica Template Method

The replica technique as far been adopted by ceramic materials researchers in the fabrication of non-porous ceramics with high porosity, pores interconnectivity and open cell walls. The fabrication process is initiated by impregnating a typical template with ceramic slurry and precursor solution. In view of developing error-free macro-porous ceramics, it is of great importance that the template in use must have a uniformly distributed open cells and a tolerable ductility level co-occurring with rapid shape recovery potential. One of such candidates with the requisite properties needed for the replica template method is the polymeric sponge which has accounted for a great proportion of macro-porous ceramics developed using this technique.

Focusing on the fabrication of reticulated porous SiC ceramics with bentonite addition, Soy et al. utilized the replica processing route to develop a highly porous ceramic foam for liquid metal infiltration using polyurethane sponge after which samples were fired at 500°C for 30min to burn out the polyurethane sponge (Soy et al., 2011). The authors observed the formation of a reformed microstructure by the over-sintering process with the presence of micro pores within the cell walls as a substitute for shrinkage which is suitable for geometry control and liquid metal infiltration.

In their investigation, Xue & Wang prepared porous SiC ceramics from waste cotton linter by reactive liquid Si infiltration technique (Xue & Wang, 2010). Cleaned cotton linters were first subjected to the carbonization process under the N_2 atmosphere after which the porous carbon output was infiltrated with liquid silicon in a vacuum oven at 1550°C. A rise in the bending strength and fracture toughness of the resulting SiC/Si composites with increasing infiltration/reaction time of liquid Si in porous carbon was reported. The authors ascribed the development of porous SiC with best operational properties such as allowable open porosity, bending strength and fracture toughness to the efficient control of the Si removing time. Similarly, Dhiman et al. successfully synthesized porous SiC elements with natural wood as starting materials by exploring the shape memory approach. Relative to the density of the bulk SiC ($3.21g/cm^3$), the density of the porous samples was around 6 times lesser which indicates the preservation of the porous structure and suitability of the fabrication technique (Dhiman et al., 2011).

Sacrificial Fugitives Method

Besides the fabrication routes discussed in previous sections, porosity can be imbued into ceramic materials by introducing the optimal quantity of pore formers into the ceramic matrix which are burned out or evaporated during the sintering process. Generally, pore formers can be categorized into; (i) synthetic organics (polymer beads, organic fibres, etc.), (ii) natural organics (potato starch, cellulose,

cotton etc.), (iii) metallic and inorganic materials (nickel, carbon, fly ash, glass particles, etc.), and (iv) liquid (water, gel, emulsions, etc.) (Ohji & Fukushima, 2012).

As highlighted in the review report of Eom et al. (2013), both natural and synthetic organics gets eliminated out of the host ceramic matrix through heat treatment application between the range of 200°C and 1000°C, liquids are extracted through freeze drying and sublimation, carbon SiO₂ templates are removed by oxidation and chemical leaching respectively and lastly, salts are often extracted by leaching using water (Eom et al., 2013). It is worthy of note that the porosity of the porous ceramics fabricated using this method can be decently tailored with varying formulations of the pore former and the particulate geometry of the pore former. The nature and porosity parameters of typical pore forming agents are presented in Table 1.

Table 1: Typical examples of sacrificial fugitives (Eom et al., 2013).

| Template used | Cell structure | Cell size (µm) | Porosity (%) |
|---|-------------------|----------------|--------------|
| Synthetic organics | | | |
| Nylon | Open cells | | |
| Poly (methacrylate-co-ethylene glycol dimethacrylate) | Open/closed cells | 5-18 | 15-88 |
| Polymethyl methacrylate | Open/closed cells | 1-150 | 60-85 |
| Poly (ether-co-octene) | Open cells | 3-14 | 78-83 |
| Natural organics | | | |
| Wax | Open cells | 100-700 | 45-65 |
| Dextrin | Open/closed cells | | |
| Yeast | Open cells | 200-500 | 61-64 |
| Liquids | | | |
| Water | Open cells | 34-147 | ~87 |
| Camphene | Open cells | 12-30 | 74-86 |
| Salt | | | |
| NaCl | Open cells | 10-40 | |
| Metal/ceramics | | | |
| Carbon | Open cells | 1-20 | 30-60 |
| Silicon | Open cells | 10-150 | 54-64 |
| SiO ₂ | Open cells | 0.5-2 | |

Research exploration of Markovska et al. indicated the development of porous mullite ceramic from waste aluminum oxide and waste rice husk with the latter doubling as the silica source as well as the burning out additives (Markovska et al., 2013). Focusing, on a reformative approach in the choice of sacrificial templates, Mohanta et al. reported a cost effective method for producing porous alumina ceramics by using rice husk (RH) as a pore former and sucrose as both a binder as well as a pore former (Mohanta et al., 2014). Sucrose was introduced into the milled powders of Al₂O₃ and RH using 20wt% sucrose solution (12wt% sucrose on dry weight basis) after which samples were compacted and subjected to an optimized burn out process. In closing with their findings, using this method indicated a successful development of porous alumina ceramics with isolated and/or interconnected pores with porosity in the range of 26-66vol%.

Using NaCl as pore former, Hu et al. investigated the development of porous Ti₂AlC with tailored porosity and pore size. NaCl powders of varying particle sizes were ball milled with Ti₂AlC and cold pressed in a die at a pressure of 800MPa (Hu et al., 2012). Thereafter, NaCl particles were eliminated by soaking the green bodies in water overnight after which the samples were then subjected to pressureless sintering process under argon ambience at 1400°C for 4h. By varying the particle size and volume

fraction of NaCl pore former, samples with homogeneously distributed pores within the Ti_2AlC matrix were developed and a broad range of porosity from 10 to 71vol% was recorded in the samples.

Direct Foaming Method

This technique employs the use of blowing agents into a ceramic suspension to create stabilized foam, dried and thereafter sintered to obtain a fully integrated macroporous ceramic structure. Eom et al. (2013) highlighted the two basic categories of blowing agents to be the physical and chemical blowing agents (Eom et al., 2013). The chemical blowing agent undergo a chemical reaction resulting in the production of gaseous products whereas, the bubble/foam-making process is reversible for the physical blowing agent and the process does not give way for any chemical reaction to take place.

Using a polycarbosilane preceramic polymer and a chemical blowing agent (azodicarbonamide), Fukushima & Colombo produced macro-cellular porous silicon carbide foams (Fukushima & Colombo, 2012). Mixture of polycarbosilane (PCS) with the blowing agent was prepared by the ball milling process and the mixture was subjected to the foaming process near the melting point of PCS and 250-260°C. Subsequently, curing of the foamed PCS occurred alongside pyrolysis at 200°C and 1000°C respectively after which the open macro-cellular ceramic components were foamed. With porosity ranging from 59 to 85vol% and cell size ranging from 416 to 1455 μ m, the authors concluded that the process can be efficiently used in producing silicon carbide based foam with decently controlled pore network and porosity level.

Current and Potential Areas of Application

According to Kumar & Kim, it was disclosed that the consideration of porous ceramics in a multiplicity of applications such as diesel particulate filters (DPFs), molten metal and hot gas filters, vacuum checks, preforms for metal-matrix composites, membrane supports for hydrogen separation, light weight structural materials, porous bio implants etc. can be linked with the unique property combination of, controlled permeability, good thermal shock resistance, low density, appreciable mechanical strength and chemical stability at elevated temperatures (Kumar & Kim, 2010). This section therefore covers the various applications of macro-porous ceramics with respect to previous and ongoing investigations.

Advancement of Porous Ceramics in Filtration Processes

Porous ceramic materials offer a great deal of advantages in filtration application effected to remove contaminants within a range of several micrometers down to the nanometer sizes. Wakita, explained that due to the better flux capability, wider pore network and reliable durability of ceramic filters over the organic hollow filter counterparts, health debilitating bacteria such as bacillus coli as well as waste water suspensions are efficiently eliminated when this category of filters are utilized during water purification process (Wakita, 2010). Research exploration of Hammel et al. highlighted three typical filtration modes as shown in Figure 2 (Hammel et al., 2014).

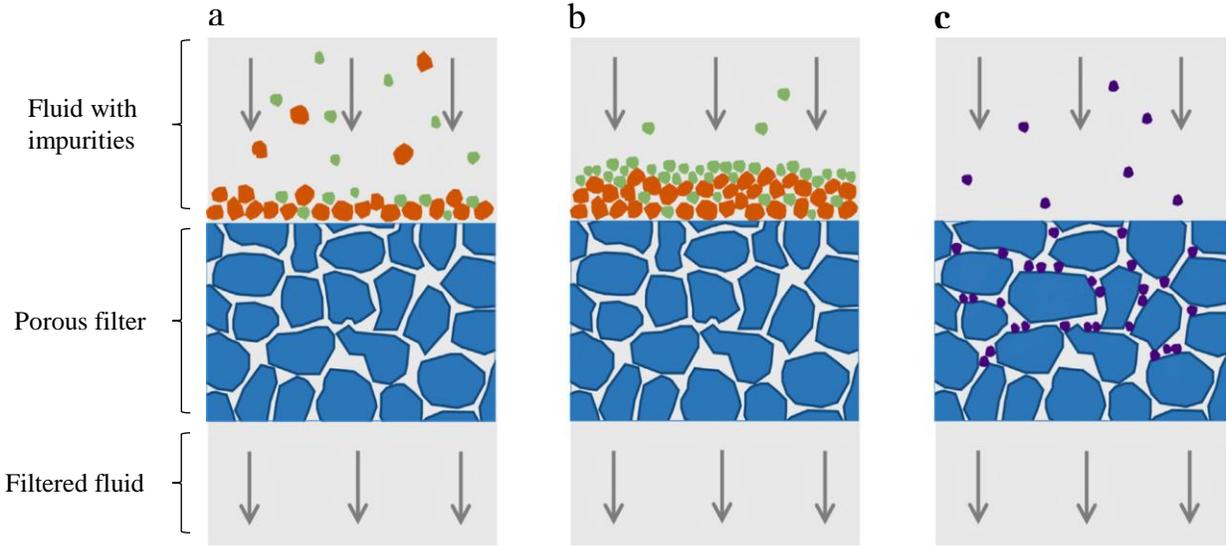


Figure 2: Representative images of the three modes of filtration: (a) surface filtration where impurities are captured at the filter due to size exclusion, (b) cake filtration where the built-up impurities act to further filter the fluid, and (c) depth filtration where the impurities are captured in the interior of the filter (Hammel et al., 2014).

In their bid to discover the most sustainable water treatment technique, Sobsey et al. carried out a critical evaluation on the point-of-use (POU) water treatment technologies ranging from: (i) chlorination with safe storage, (ii) combined coagulant-chlorine disinfection systems, (iii) SODIS, (iv) ceramic filter and (v) biosand filter (Sobsey et al., 2008). Out of the technologies reviewed, the authors proffered both the ceramic and biosand filters as having great potentials in water purification systems owing to the improved water quality production, effective performance long after implementation and high year's post-implementation usage rates of filters.

A breakdown of hot gas filtration applications is presented in Table 2. Heidenreich revealed an asymmetric high density ceramic structured with a thin layer membrane having tiny pores above the support material (Heidenreich, 2013). Surface filtration was effectively achieved with the membrane which can be linked with the reliable behavior of the filters and their lifetime. More so, the report of Cummer & Brown concluded that the collection efficiency of highly dense ceramic filter components approached nearly a 100% (Cummer & Brown, 2002).

Table 2: Breakdown of hot gas filtration applications (Heidenreich, 2013).

| | |
|--|---|
| Coal gasification | Smelting processes |
| Coal combustion | Metal production |
| Biomass gasification | Metal recycling (e.g. Aluminum recycling) |
| Biomass pyrolysis | Glass industry |
| Biomass combustion | Cement industry |
| Refineries | Steel industry |
| Low-level radioactive waste incineration and pyrolysis | Chemical industry (e.g. production and recovery of catalysts) |
| Waste incineration and pyrolysis | Production of metal oxide powders, pigments and nanoparticles |

With the primary aim of improving combustion efficiency and minimize emission of air pollutants, industries have adopted the utilization of ceramic filters in diesel engines to trap particulate matter in the exhaust gas stream. Meanwhile, owing to more stringent environmental legislations, the demand for diesel particulate filters (DPFs) is expected to further increase across the globe (Shyam et al., 2008). Investigation by Sundaram et al., highlighted both health and environmental issues to be the drivers for the development of next-generation efficient ceramic filters whose implementation as DPFs can substantially reduce particulate matter (PM) and nitrogen oxides (NO_x) emissions (Sundaram et al., 2013).

Porous Ceramics for Thermal Insulation

Considering the light weight and low thermal conductivity properties of ceramic materials, Hammel et al. emphasized on the remarkable high temperature and chemical stability of ceramic materials in general which makes them fit for high temperature chemical processes (Hammel et al., 2014). They further considered that the operational performance of insulators is greatly dependent on their resistance capacity to heat flow through all forms of thermal transport (conduction, convection and radiation).

In view of developing such decent ceramic thermal insulator, several fabrication methods have been explored thus far, yet, the outcome has been less favorable. However, the development of highly porous ceramic foams with mechanical strength using the freeze casting method was reported by Fukushima (Fukushima, 2013). Capitalizing on the much lower thermal conductivity of a porous ceramic as against its dense bulk, Li & Li fabricated porous Y₂SiO₅ ceramic having a low thermal conductivity via Tert-butyl alcohol (TBA) based freeze casting (Li & Li, 2012). The authors reported a decline in compression strength from 23.2 to 3.1MPa and thermal conductivity reduction in porous Y₂SiO₅ ceramic (0.05W/mK for 57% porosity) as compared to that of the dense bulk ceramic.

Through the utilization of the freeze casting processing route, Hu et al reported the investigation of porous YSZ ceramic with unidirectionally aligned pore channel size. (Hu et al., 2010). With low thermal conductivities within a range of 0.06W/mK and 0.30W/mK highlighted in their work, the porous YSZ ceramics developed by this method will offer distinct application in thermal insulation systems since the high porosity at the end along the channel direction will give an appreciable thermal insulation efficiency while the other end with a denser structure will provide optimal strength for load bearing.

Porous Ceramics for Bone Tissue Engineering Applications

In recent years, bone tissue engineering has offered a promising approach for bone repair and construction in conditions such as anhrthritis, osteoporosis, traumatic musculoskeletal injuries, spinal injuries and spinal deformities (Liu et al., 2013; Vlasea et al., 2013). Ghazanfari & Zamanian, reported the effect of nano-alumina content on phase transformation, microstructural and mechanical properties of hydroxyapatite/nano-alumina (HA/nAl₂O₃) nanocomposite scaffolds (Ghazanfari & Zamanian, 2013). From their result, increasing the Al₂O₃ content led to increasing the pore size and the compressive strength of the scaffolds.

In their work, Tripathi & Basu indicated the needed criteria for the development of porous scaffold which can meet up with the compatibility requirements of the intended application (Tripathi & Basu, 2012). These includes; (a) biocompatibility, which enables the cell growth, surface attachment and proliferation, (b) the component should promote strong bone bonding, resulting in osteoconduction and osteoinduction, (c) rate of new tissue formation and biodegradability should match with each other, (d) the mechanical strength of the scaffolds should be adequate enough to provide mechanical constancy in load bearing sites prior to regeneration of new tissue and (e) porous structure and pore size of more than 100µm for cell penetration, tissue in growth and vascularization.

Porous Ceramic Preforms for Fabrication of Ceramic Based Composites

In their application based review, Mortensen and Liorca (2010) revealed three basic inducements promoting rapid development of metal matrix composites (MMCs) to be; (i) the potentials of MMC in upgrading the boundaries drawn by the intrinsic properties of monolithic metals and ceramics, (ii) fabrication of MMC is the only way of incorporating an optimal content of oxide or carbide into a metal and (iii) introduction of fine ceramic particles in metallic matrices gives way for the properties of the ceramic materials to be exhibited in the developed MMC.

Hammel et al. emphasized on the importance of controlling the distribution of pores in the preforms owing to the higher reliability potential of preforms with regular pore spacing (Hammel et al., 2014). Furthermore, the preform must have interconnected pore structure to ease molten metal infiltration since blocked pores will inhibit the infiltration and thereby reducing porosity level within the ceramic component. Roy et al. fabricated porous alumina preforms by pyrolysis of cellulose fibres used as a pore-forming agent and subsequent sintering of alumina particles (Roy et al., 2011). Afterwards, both the micropores and macropores within the ceramic walls were infiltrated by the liquid metal.

So far, it has been established that majority of metal-ceramic composites exhibit superior fracture toughness and damage tolerance when compared to monolithic ceramics (Chang et al., 2010). Consolidating on this advantage, the authors investigated gel-cast Al_2O_3 foams with spherical interconnected open porosity which were pressurelessly infiltrated using Al-8wt% Mg alloy. The high strength recorded was attributed mainly to the preferential propagation of cracks in the ceramic phase, whilst good metal-ceramic interfacial bonding, metal bridging through plastic deformation and the crack deflection.

Conclusion

With a view to documenting the recent studies conducted thus far in the processing and application of porous ceramic components, this review comprehensively reveals both reliable and affordable processing techniques as well as potentials of porous ceramic's usage in engineering and medical fields. The article presents both state of the art technologies as well as economically viable methods such as the sacrificial fugitives method wherein organic matters can be used as pore formers for lab scale development of porous ceramic materials. More so, through the utilization of porous ceramics as diesel particulate filters, a substantial reduction in particulate matter emission from internal combustion engines powered by diesel fuel has been achieved. In a similar way, ample success has been achieved in the field of bone tissue engineering by employing porous ceramic scaffolds as replacements for fractured bones.

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