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# Processing and properties of polysiloxane-derived porous silicon carbide ceramics using hollow microspheres as templates

Jung-Hye Eom[a], Young-Wook Kim[a,*], In-Hyuck Song[b], Hai-Doo Kim[b]

[a] *Department of Materials Science and Engineering, The University of Seoul, 90 Jeonnong-Dong, Dongdaemoon-Ku, Seoul 130-743, Republic of Korea*
[b] *Ceramic Materials Group, Korea Institute of Machinery and Materials, Changwon, Gyeongnam 641-010, Republic of Korea*

## Abstract

Porous SiC ceramics were synthesized from carbon-filled polysiloxane using hollow microspheres as sacrificial templates. The fabrication process involved three steps: (i) the pyrolysis of polysiloxane at 1100 °C, which leads to the conversion of polysiloxane to silicon oxycarbide (SiOC); (ii) the carbothermal reduction of SiOC and the C mixture at 1450 °C, which converts the mixture to a SiC ceramic; and (iii) liquid-phase sintering of the SiC using $Al_2O_3$–$Y_2O_3$ as a sintering additive at 1800–2000 °C.

The effects of the sintering temperature and template contents on the microstructure, porosity, mechanical strength and thermal conductivity of the resulting porous SiC ceramics were examined. The typical flexural and compressive strengths of the porous SiC ceramics with ~40% porosity were ~60 MPa and ~240 MPa, respectively. The typical thermal conductivity of the porous SiC ceramics with ~70% porosity was ~2 W/m K.

*Keywords:* SiC; Porosity; Mechanical properties; Thermal conductivity

## 1. Introduction

Porous SiC ceramics are of technological interest on account of their unique characteristics, which include low density, low thermal conductivity, low thermal mass, controlled permeability, high thermal shock resistance, high surface area, and high specific strength. These properties cannot normally be achieved with their conventional dense counterparts. Hence, these types of ceramics are finding many applications including the filtration of molten metals, filtration of particles from diesel engine exhaust gases, filtration of hot corrosive gases in various industrial applications, gas-burner media, membrane supports for hydrogen separation and light-weight structural components for high temperature applications.[1–6] Since the open and closed porosity, pore size distribution and pore morphology in porous ceramics is related directly to their ability to perform a desired function in a particular application, there is a need to control these characteristics in order to achieve these superior properties.[7–9] All these characteristics are in turn strongly influenced by the processing route and templates used for producing porous ceramics.

Several processing routes for porous SiC ceramics have been developed for specific applications as well as their associated requirements of porosity, pore size and degree of interconnectivity. These manufacturing techniques are typically divided into three categories: replica techniques, sacrificial template techniques and reaction techniques. The replica method is based on the impregnation or coating of a cellular structure with a ceramic suspension or precursor solution to produce a porous SiC ceramic showing the same morphology as the original porous material. Polyurethane foams,[4] carbon foams[10] and wood[11] have been used as templates to fabricate porous SiC ceramics using this technique. The sacrificial template technique usually consists of the preparation of a composite comprising a matrix of SiC particles and a dispersed sacrificial phase that is ultimately extracted to generate pores within the microstructure. Salt compacts,[12] polymer microbeads,[7] yeast[13] and nylon filter[14] have also been used as templates to fabricate porous SiC ceramics using the sacrificial template technique. Reaction methods involve oxidation bonding techniques,[15] siliciding techniques[16] and carbothermal reduction techniques.[17] Oxidation bonding techniques involve the heating of SiC powder compacts in air so that the SiC particles are bonded to each other by an oxidation-derived $SiO_2$ glass[15] or by oxidation-derived mullite with the addition of $Al_2O_3$ addition.[18] Siliciding techniques involve a chemical reaction between wood-derived carbon foams and

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[*] Corresponding author. Tel.: +82 2 2210 2760; fax: +82 2 2215 5863.
*E-mail address:* ywkim@uos.ac.kr (Y.-W. Kim).

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molten Si.[16] The carbothermal reduction techniques involve the carbothermal reduction of $SiO_2$ or polysiloxane-derived SiOC by carbon.[17,19] There are no reports of the mechanical strength and thermal conductivity of porous SiC ceramics processed by carbothermal reduction.

In this study, porous SiC ceramics were fabricated by the carbothermal reduction of a polysiloxane-derived SiOC with hollow microspheres being used as sacrificial templates. One of the potential advantages of using polysiloxane (a preceramic polymer) for fabricating porous SiC ceramics is the utilization of low-cost polymer processing such as extrusion. The use of hollow microspheres as sacrificial templates is also advantageous over other templates such as PMMA-PEG microbeads because of the lower amounts of gaseous by-products generated during pyrolysis and the lower cost than bulk microbeads. The effects of the sintering temperature and template contents on the microstructure, porosity, mechanical strength and thermal conductivity of the resulting porous SiC ceramics were investigated.

## 2. Experimental procedure

The raw materials used included: a polysiloxane (YR3370, density = 1.036 g/cm$^3$, GE Toshiba Silicones Co., Ltd, Tokyo, Japan), a carbon black (Corax MAF, Korea Carbon Black Co., Ltd., Inchon, Korea), SiC (Ultrafine grade, Betarundum, Ibiden Co. Ltd., Ogaki, Japan), hollow microspheres (461DE20, Expancel, Sundsvall, Sweden), $Al_2O_3$ (AKP30, Sumitomo Chemical Co., Tokyo, Japan) and $Y_2O_3$ (H.C. Starck GmbH & KG, Goslar, Germany). The polysiloxane was solid at room temperature. The SiC was added as an inert filler, and the $Al_2O_3$ and $Y_2O_3$ were added as sintering additives. The inert filler was added for both decreasing shrinkage during sintering and increasing strength of porous SiC ceramics.[20] The microspheres are hollow poly(methyl methacrylate) spheres with diameters ranging from 15 μm to 25 μm.

Five batches of powder were prepared and the content of the microspheres in those batches ranged from 0 wt% to 10 wt% (Table 1). An example of sample notation is as follows: PS5 denotes a sample containing 5 wt% hollow microspheres as the sacrificial templates. All individual batches were mixed in ethanol for 3 h using SiC balls and a polyethylene jar. The milled powder was dried and uniaxially pressed into rectangular bars at 28 MPa. The compacts formed were cross-linked by heating them to 200 °C in air. The cross-linked samples were pyrolyzed at 1100 °C for 1 h at a heating rate of 1 °C/min in argon. The pyrolysis was performed in a horizontal tube furnace (MSTF1500, Myungsung Engineering Inc., Korea) equipped with an alumina tube of 70 mm inner diameter. The heat-treatment allows for the conversion of polysiloxane to silicon oxycarbide in the specimens.[21] The composition of SiOC after pyrolysis at 1400 °C was $SiO_{1.50}C_{0.68}$.[22] The pyrolyzed specimens were further heat-treated in argon to 1450 °C for 1 h at a heating rate of 10 °C/min and subsequently annealed at 1800–2000 °C for the liquid-phase sintering of SiC using $Al_2O_3$ and $Y_2O_3$. The heat-treatment at 1450 °C and annealing at 1800–2000 °C were performed in a graphite furnace (1000-3560-FP20, Thermal Technologies Inc., USA).

The bulk density of the porous ceramics was calculated from the weight-to-volume ratio of the samples. The open porosity and pore size distribution were measured using a mercury porosimetry (AutoPore IV 9500, Micromeritics, USA). The cell and grain morphology was observed by scanning electron microscopy (SEM, S4300, Hitachi Ltd., Japan). Using Cu Kα radiation, X-ray diffractometry (XRD) was performed on ground powders on a diffractometer (D8 Discover, Bruker AXS GmbH, Germany). The porosity was determined from the bulk density to true density ratio. For flexural strength measurements, bar-shaped samples were polished to a normal size of 4 mm × 5 mm × 30 mm. Bend tests were performed at room temperature using a four-point method with an inner and outer span of 10 mm and 20 mm, respectively, on an Instron machine (4465, Instron Co., Ltd., USA). The compressive strength was measured at a crosshead speed of 0.5 mm/min on 4 mm × 4 mm × 8 mm-sized samples. The thermal conductivity was measured on 8 mm × 8 mm × 2 mm-sized plate-shaped specimens using a laser flash method (LFA 437, MicroFlash$^{TM}$, Netzsch-Geratebau GmbH, Germany).

## 3. Results and discussion

### 3.1. Processing

The general reaction for synthesizing SiC from a mixture of polysiloxane and carbon black is reported elsewhere.[23] The

Table 1
Batch composition of porous SiC ceramics

| Sample | Batch composition (wt%) | | | | |
|---|---|---|---|---|---|
| | Polysiloxane[a] | Carbon black[b] | SiC[c] | Hollow microsphere[d] | Sintering additive |
| PS0 | 56.2 | 8.8 | 30 | 0 | 3% $Al_2O_3$ + 2% $Y_2O_3$ |
| PS3 | 53.6 | 8.4 | 30 | 3 | 3% $Al_2O_3$ + 2% $Y_2O_3$ |
| PS5 | 51.9 | 8.1 | 30 | 5 | 3% $Al_2O_3$ + 2% $Y_2O_3$ |
| PS8 | 49.3 | 7.7 | 30 | 8 | 3% $Al_2O_3$ + 2% $Y_2O_3$ |
| PS10 | 47.5 | 7.5 | 30 | 10 | 3% $Al_2O_3$ + 2% $Y_2O_3$ |

[a] YR3370, GE Toshiba Silicones Co. Ltd., Tokyo, Japan.

[b] Corax MAF, Korea Carbon Black Co., Ltd., Inchon, Korea.

[c] Ultrafine, Betarundum, Ibiden co. Ltd., Ogaki, Japan.

[d] 461DE20, Expancel, Sundsvall, Sweden.

reaction occurred in two steps: (i) the pyrolysis of polysiloxane at 1100 °C, which involves the loss of the organic material and also leads to the conversion of polysiloxane to an amorphous Si–O–C material (SiOC) and (ii) the carbothermal reduction of SiOC and C at 1450 °C, which converts the mixture to a SiC ceramic with the evolution of gaseous CO. Further heat-treatment of the specimens at 1800–2000 °C with the aid of $Al_2O_3$–$Y_2O_3$ led to the liquid-phase sintering of SiC that had been synthesized by carbothermal reduction, and added as an inert filler.

Fig. 1 shows XRD results of some selected specimens. As shown in Fig. 1, SiC was successfully synthesized as a result of carbothermal reduction. No other crystalline peaks except β- and α-SiC were observed in all specimens, indicating that the carbothermal reduction has been completed under the present composition and reaction conditions. XRD analysis of the other specimens showed that the 1800 °C-sintered specimens showed no phase transformation, i.e., consisted of β-SiC. However, the 1900 °C- and 2000 °C-sintered specimens showed the presence of α-SiC, indicating the occurrence of the β → α phase transformation of SiC during sintering. β-SiC was the major phase in the 1900 °C-sintered specimens whereas α-SiC was a major phase in the 2000 °C-sintered specimens.

Figs. 2 and 3 show typical fracture surfaces of the porous SiC ceramics. These figures show evidence that fine and well-distributed open cells, and that porous struts in the cellular structure had also been produced. The morphology of the primary cells (cells replicated from hollow microspheres) was almost spherical, indicating that the shape of hollow microspheres is retained in the polysiloxane–carbon black–SiC–$Al_2O_3$–$Y_2O_3$ compact up to its decomposition temperature. The cell size was smaller than 20 μm, and there were no large voids in the bulk materials when up to 8 wt% of the microsphere was loaded and sintered at temperatures lower than

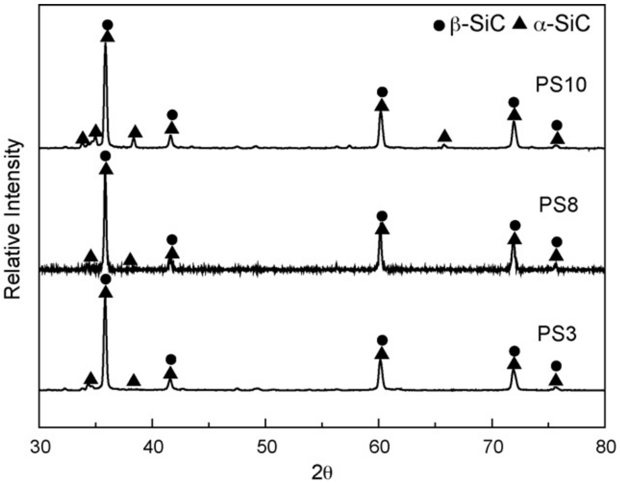

Fig. 1. XRD patterns of porous SiC ceramics sintered at 1900 °C for 1 h in argon (refer to Table 1).

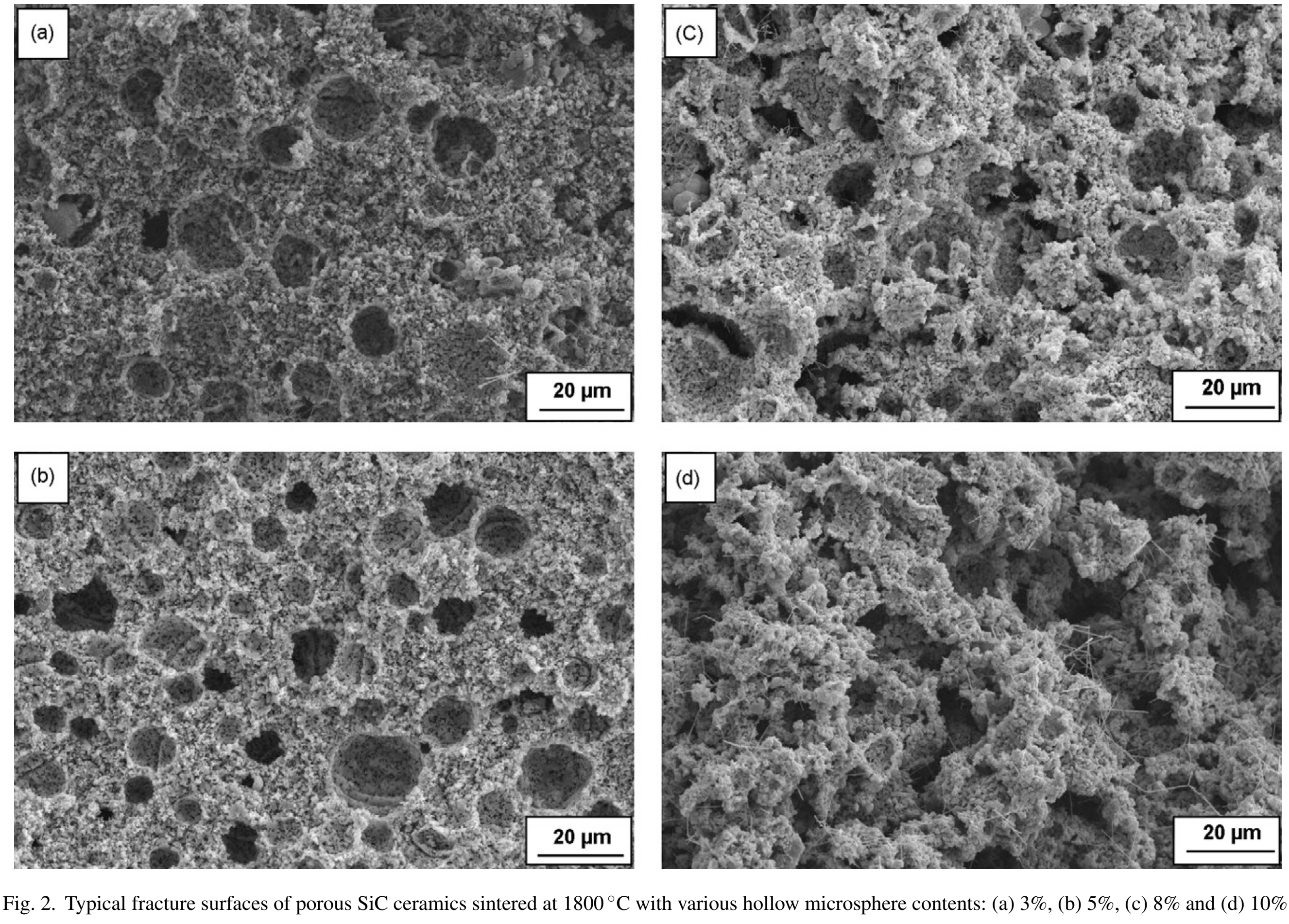

Fig. 2. Typical fracture surfaces of porous SiC ceramics sintered at 1800 °C with various hollow microsphere contents: (a) 3%, (b) 5%, (c) 8% and (d) 10%.

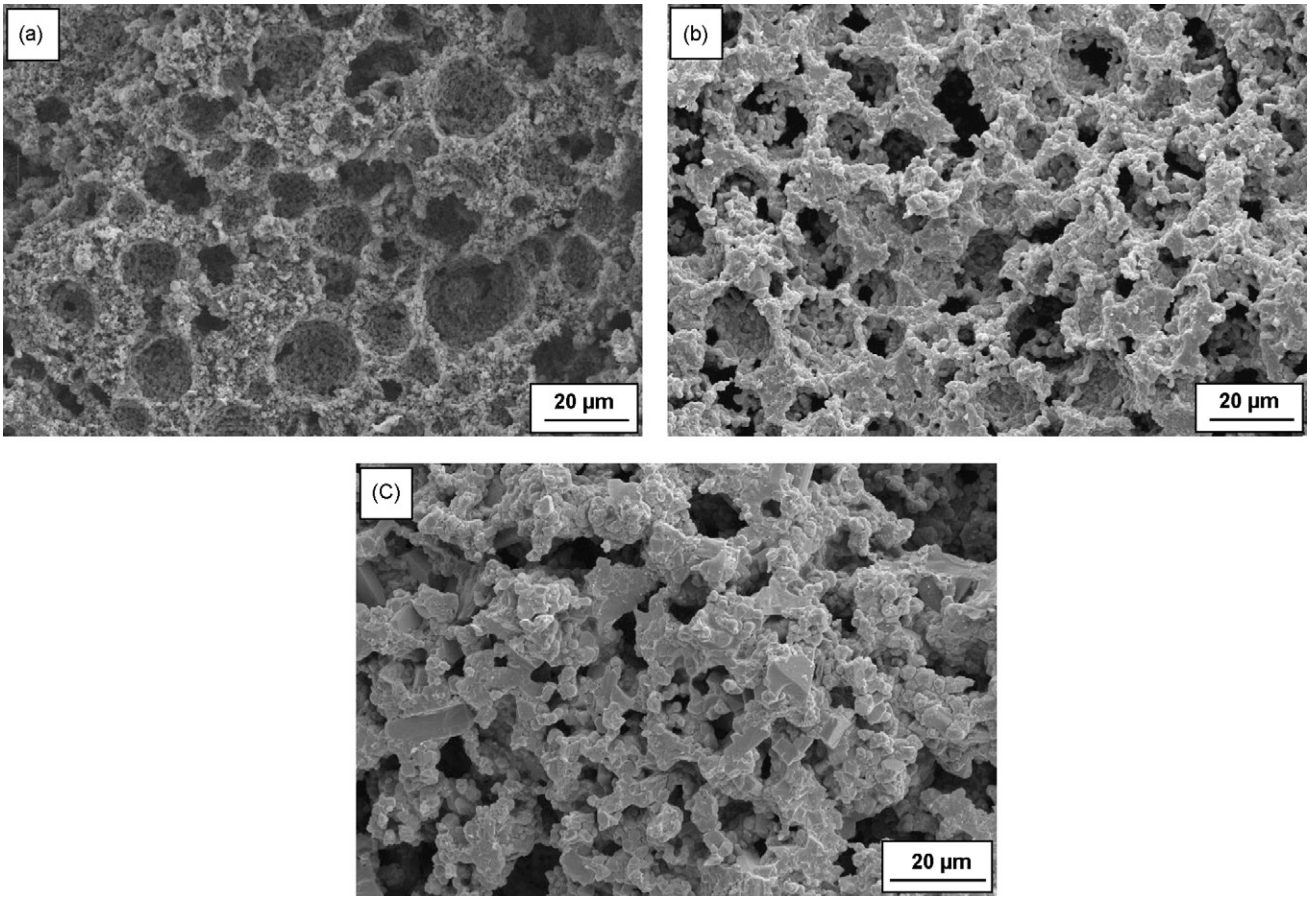

Fig. 3. Typical fracture surfaces of porous SiC ceramics sintered at various temperatures with 5 wt% hollow microspheres: (a) 1800 °C, (b) 1900 °C and (c) 2000 °C.

2000 °C. The cell morphology changed from spherical to irregular as the microsphere content was increased in the porous SiC ceramics. This change was due to the greater opportunity for contact between the microspheres in the compacts (see Fig. 2(d)). As shown in Fig. 3, an increase in the sintering temperature also led to a change in the pore morphology from spherical to irregular because of the enhanced mass transport and grain growth at the higher temperature. It is interesting that the PS3, PS8 and PS10 specimens (Fig. 2) contained needle-like SiC whiskers that had been grown *in situ*. The SiC whiskers were grown by a vapor phase reaction between SiO and CO during the carbothermal reduction of SiOC.[24] The *in situ* grown SiC whiskers were also observed in porous SiC ceramics using microbeads as the sacrificial templates.[17]

Fig. 4 shows the typical grain morphology of porous SiC ceramics. The grain size increased and the grain morphology changed from small, equiaxed grains to large, facetted grains, as sintering temperature was increased from 1800 °C to 2000 °C. The 1800 °C-sintered specimen contained a mixture of large (>1 μm) and small (<0.5 μm) grains (Fig. 4(a)). The particle size of the SiC filler was ~0.3 μm. Therefore, the large grains observed grew from the added filler and the small grains were synthesized from carbothermal reduction during processing. The large grains grew further with increasing temperature and no small grains were observed in the specimens sintered at 1900 °C, indicating the growth of large grains occurs through solution-reprecipitation at the expense of the small grains. Further grain growth and the faceting of grains were observed in the specimens sintered at 2000 °C (Fig. 4(c)). The average grain size of the 2000 °C-sintered specimen was ~2 μm. The faceting of the grains was attributed to the $\beta \rightarrow \alpha$ phase transformation of SiC.[25]

### 3.2. Properties

Fig. 5 shows the porosity of the porous SiC ceramics as a function of the hollow microsphere content and the sintering temperature. The porosities of the porous SiC ceramics ranged from 60% to 77% when sintered at 1800 °C and from 44% to 71% when sintered at 2000 °C. It should be noted that these ranges depended on both the microsphere content and the sintering temperature. The porosity varied directly with the microsphere content for all specimens observed, i.e., the higher the microsphere content, the higher the porosity. In contrast, a higher sintering temperature led to a lower porosity due to the enhanced densification at the higher temperature.

Fig. 6 shows the variation of pore size distribution from 0.003 μm to 30 μm with varying sintering temperature. The pore size distribution of PS5 specimen changed from bimodal in 1800 °C-sintered specimen to unimodal in 2000 °C-sintered specimen. Open porosity decreased from 59.6% to 48.1% with increasing the sintering temperature from 1800 °C to 2000 °C.

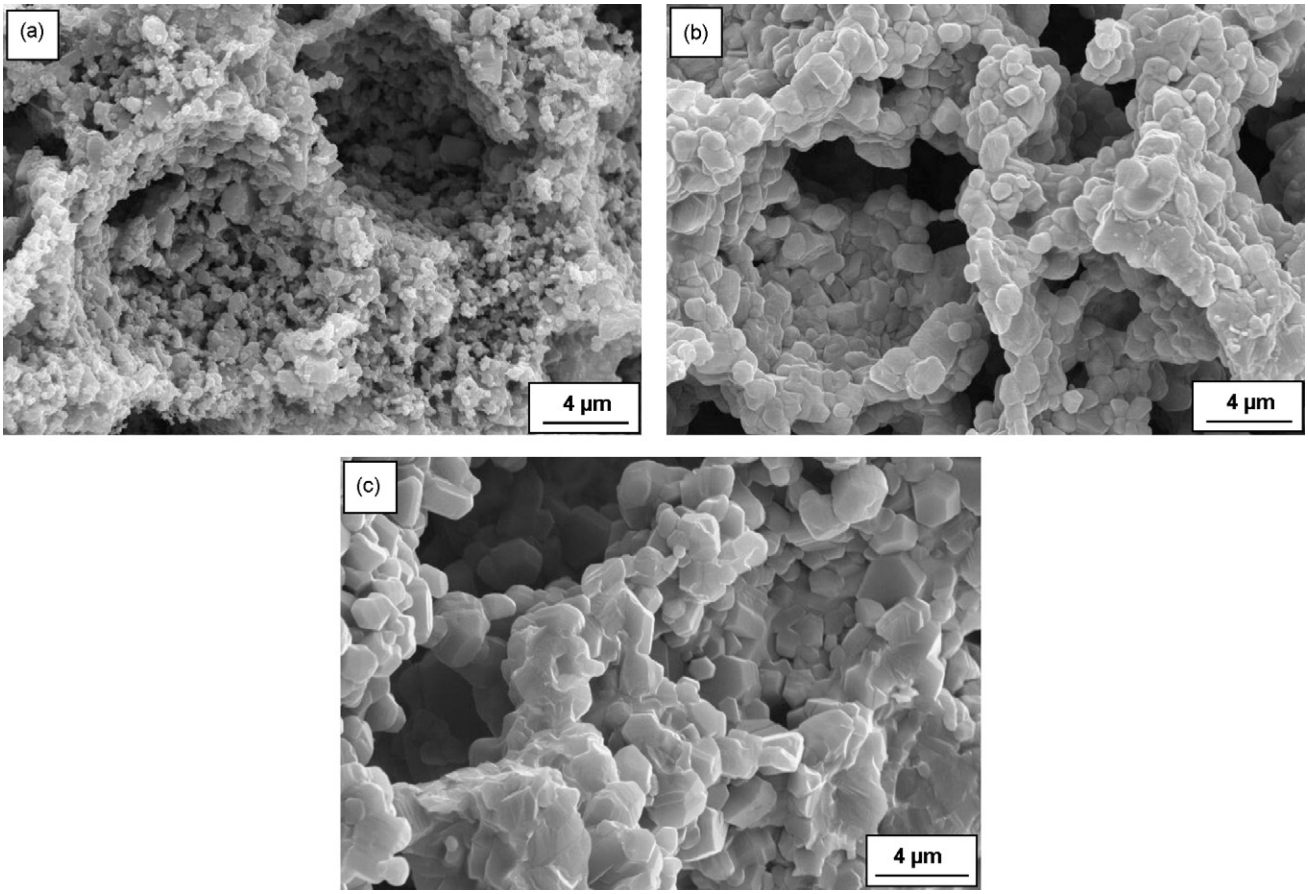

Fig. 4. Typical grain morphology of porous SiC ceramics sintered at various temperatures with 8 wt% hollow microspheres: (a) 1800 °C, (b) 1900 °C and (c) 2000 °C.

The volume fraction of submicron-size pores decreased significantly with increasing sintering temperature, owing to the densification through liquid-phase sintering. In contrast, the volume fraction of micron-size pores increased with increasing sintering temperature, owing to the grain coarsening. Lin and Tsai[26] also found that pore size increased with increasing sintering temperature. The porosities calculated from the bulk density to true density ratio of PS5 specimens sintered at 1800 °C and 2000 °C were 61.1% and 52.0%, respectively. Thus, the porosities calculated were comparable with those measured from mercury porosimetry, indicating that most of the pores in PS5 specimen were open.

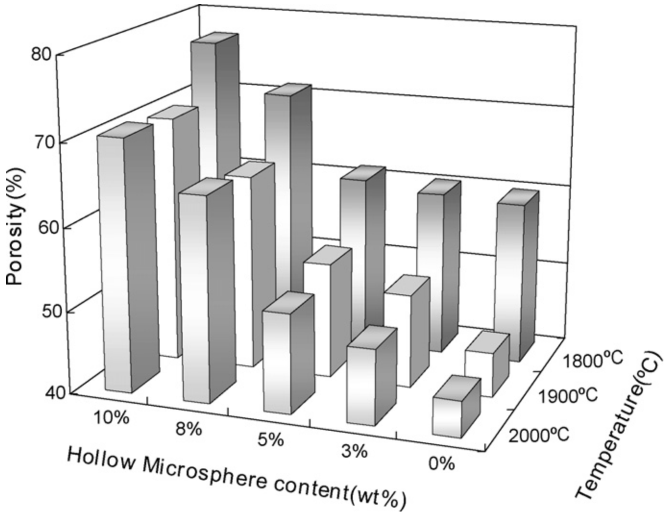

Fig. 5. Effects of the hollow microsphere content and the sintering temperature on porosity.

Figs. 7 and 8 show the flexural and compressive strengths as a function of the porosity for the porous SiC ceramics. Both strengths decreased with increasing porosity. This tendency has been observed in many other porous ceramics[4,15,23] and was attributed to the higher probability of pore coalescence at the higher porosity under load. The pore coalescence leads to a larger defect size, resulting in a lower strength. From these results, it was difficult to determine a trend in the sintering-temperature dependency of both flexural and compressive strengths. It appears that the flexural and compressive strengths are influenced preferentially by the porosity rather than by the sintering temperature. However, the sintering temperature influenced the porosity of the porous SiC ceramics, as shown in Fig. 5. Therefore, the sintering temperature indirectly affected the strength of the porous SiC ceramics. Typical flexural strengths of the porous SiC ceramics with ∼40% and 50% porosity were ∼60 MPa and 45 MPa, respectively. Typical compressive strengths of the porous SiC ceramics with ∼40% and 50% porosity were ∼240 MPa and 175 MPa, respectively. Flexural strengths of ∼10 MPa at ∼50% porosity[27] and

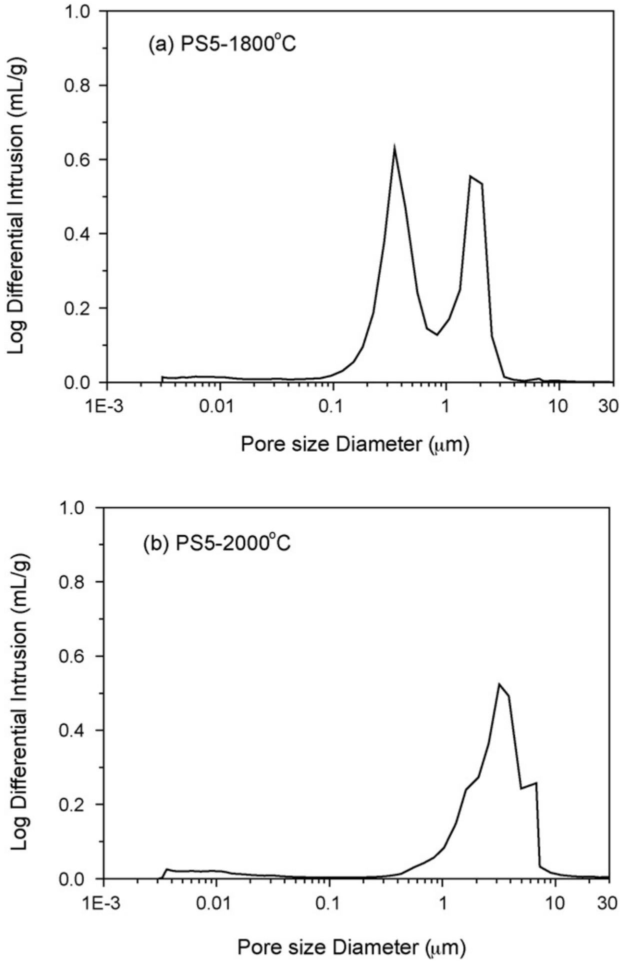

Fig. 6. Pore size distribution of porous SiC ceramics sintered at (a) 1800 °C and (b) 2000 °C.

~28 MPa at ~44% porosity[28] have been reported in reaction-bonded porous SiC ceramics and 17 MPa at ~61% porosity in sintered porous SiC ceramics.[13] A compressive strength of ~160 MPa was also reported at 40% porosity in porous SiC ceramics prepared using polymer microbeads as the sacrificial template.[29] The superior strength of the porous SiC ceramics was attributed to both the lack of macroscopic defects (Fig. 2) and the presence of a rigid strut with a well-developed grain structure (Fig. 4(b and c)). These results suggest that hollow microspheres are suitable templates for preparing high-strength, open-cell SiC ceramics by carbothermal reduction and a subsequent sintering process.

Fig. 9 shows the thermal conductivity of the porous SiC ceramics. The thermal conductivity decreased with increasing porosity. A low thermal conductivity of ~2 W/m K was obtained at ~70% porosity. The thermal conductivity of the dense SiC ceramics sintered with $Y_2O_3$–$La_2O_3$ ranged from 169 W/m K to 206 W/m K.[30] BeO-doped SiC showed a thermal conductivity as high as 250 W/m K.[31] The thermal conductivities of $B_4C$-doped SiC and $Al_2O_3$–$Y_2O_3$-doped SiC were reported to be 120 W/m K[32] and 70–90 W/m K,[33] respectively. Sigl[34] suggested that the high solubility of Al in the SiC lattice reduces the thermal conductivity of the liquid-phase sintered SiC containing $Al_2O_3$ and/or AlN additives. Therefore, the low thermal conductivity of the porous SiC ceramics fabricated in this study was attributed to the introduction of pores into the microstructure and partly to the introduction of Al into the SiC lattice.

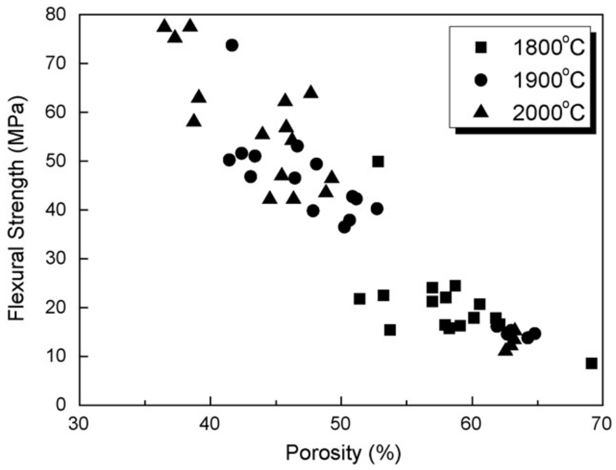

Fig. 7. Flexural strength of the porous SiC ceramics as a function of porosity.

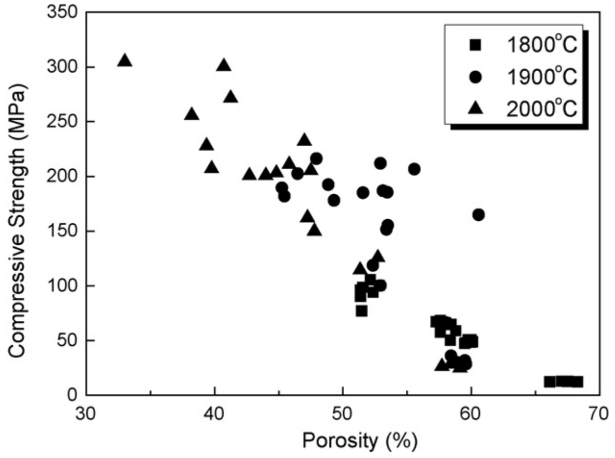

Fig. 8. Compressive strength of the porous SiC ceramics as a function of porosity.

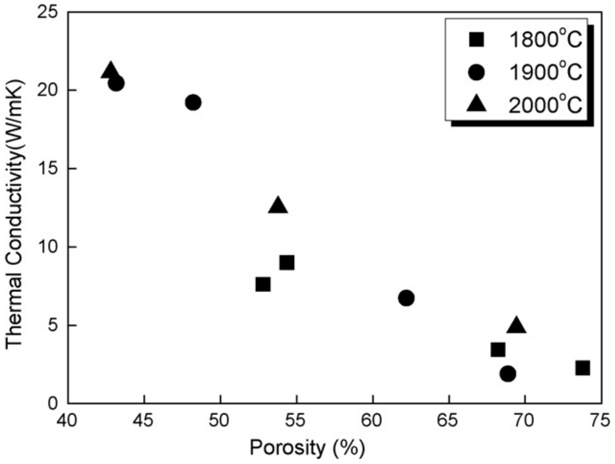

Fig. 9. Thermal conductivity of the porous SiC ceramics as a function of porosity.

## 4. Conclusions

Hollow microspheres were used successfully as sacrificial templates for producing partially interconnected, open-cell SiC ceramics from a carbon-filled polysiloxane. It was possible to adjust the porosity ranging from 44% to 77% by controlling the microsphere content and the sintering temperature. The grain morphology changed from small, equiaxed grains to large, facetted grains with increasing sintering temperature from 1800 °C to 2000 °C due to the $\beta \rightarrow \alpha$ phase transformation of SiC during sintering.

The typical flexural and compressive strengths of the porous SiC ceramics with 50% porosity were ∼45 MPa and ∼175 MPa, respectively. The thermal conductivity of the porous SiC ceramics with ∼70% porosity was as low as ∼2 W/m K. These results suggest that the proposed novel processing technique is suitable for manufacturing partially interconnected, open-cell SiC ceramics with a tailored porosity and superior properties.

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