

# Porous SiC ceramics prepared via freeze-casting and solid state sintering

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Received 30 September 2015; received in revised form 20 November 2015; accepted 25 November 2015

Available online 2 December 2015

## Abstract

Porous SiC ceramics were prepared by freeze-casting process. In order to enhance the mechanical properties of the porous SiC, poly(vinyl alcohol) (PVA) was added as binder and pore morphology controller in this work. The results indicated that high porosity (> 60%) SiC ceramics was obtained although the sintering temperature was over 2000 °C. The pore structure could be divided into two kinds: macropores generated by sublimation of large ice crystals, and micropores in the ceramic matrix caused by sublimating of small ice crystals, stacking of SiC particles, and burning out of PVA. With the increase of the sintering temperature, the specimens exhibited higher density, thus resulted in higher strength. Porous SiC ceramics sintered at 2100 °C showed a good flexural strength of 11.25 MPa with an open porosity as high as 66.46%.

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**Keywords:** D. SiC; Freeze-casting; Solid state sintering; Flexural strength; Open porosity

## 1. Introduction

Porous SiC ceramic is one of the promising candidate for the application as hot-gas or molten-metal filters, gas-burner media and catalytic supports owing to its excellent properties, including their high temperature strength, chemical stability, and oxidation resistance [1–6]. Normally, porous SiC ceramics are prepared by liquid state sintering and in situ reaction bonding process, which brings in other phases, such as alumina, yttria, mullite, and cordierite [6–10] located at the grain boundaries, resulting in the operating temperature of porous SiC ceramics is generally below 1500 °C due to the low temperature phases. As a result, these porous SiC ceramics are not suitable for the high temperature applications.

Solid-state-sintered SiC ceramics have much cleaner grain boundaries, resulting in better high temperature stability and corrosion resistance [11]. But the solid state sintering is normally applied to prepare dense SiC ceramics with high relative density (> 95%) [12–15]. Few works on porous SiC

ceramics prepared by the solid state sintering method have been reported. Liu et al. [16] fabricated the SiC foams via solid state sintering with porosity of 61–81%, but the bending strength is only 1.5–4.8 MPa.

Nowadays, freeze-casting method has aroused special interesting due to its notable advantages, especially the environmental friendly aqueous suspensions are applied. The sublimation of ice crystals, which generates pores in the ceramics, is environment-friendly and will not bring impurities into the samples [17]. Moreover, by changing the freeze conditions, the morphologies of the pores can be well controlled [18]. Zuo et al. [19] proved that the freeze-casting combining solid state sintering method was an effective way to achieve porous ceramics with widely controllable porosity and high mechanical strength.

In this work, porous SiC ceramics with high open porosity and good mechanical properties were prepared via freeze-casting combining solid state sintering. The effects of PVA content and sintering temperature on the microstructures and performances of porous SiC ceramics were discussed. The flexural strength, morphology, pore size distribution, linear shrinkage and open porosity of porous SiC ceramics were characterized.

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## 2. Experimental procedures

The starting powder was commercially available SiC powder ( $d_{50}=0.5\ \mu\text{m}$ , a-phase, FCP15C, Sika Tech., Lille-sand, Norway). B<sub>4</sub>C powder ( $d_{50}=1.0\ \mu\text{m}$ , Mudanjiang Jintangzuan Boron Carbide Co., Ltd, China) and carbon powder ( $d_{50}=0.2\ \mu\text{m}$ , Shanghai Coking & Chemical Development, Shanghai, China) were used as the sintering additives in the solid-sintering process. The weight percentages of carbon and B<sub>4</sub>C powder based on the SiC powder were 2.5 wt% and 0.5 wt%, respectively. Tetramethylammonium hydroxide (TMAH, TaixingHaoshen Chemical Trading CO., Ltd, China) was used as dispersant agent, and the weight percent of TMAH based on the SiC powder was 0.6 wt%. Poly(vinyl alcohol) (PVA, Sinopharm Chemical Reagent Co., Ltd, China) was used as binder and pore morphology controller. SiC suspensions with initial solid content of 50 wt% were prepared by mixing SiC powder with sintering additives and dispersant agent in the distilled water. The suspensions were ball-milled for 1 h at a rotate speed of 300 r/min. Further ball-milled in planetary ball mill for 3 h at the same speed was continued after adding poly(vinyl alcohol) aqueous solution (10 wt%). The ratios of PVA to the SiC powder were 2.5 wt%, 5 wt%, 7.5 wt% and 10 wt%, respectively. These mixed suspensions were named as 2.5P, 5P, 7.5P and 10P suspensions, respectively. The final solid contents of these suspensions were 44.59 wt%, 40.23 wt%, 36.65 wt%, and 33.66 wt%. Defoaming agent would be used if bubbles were generated in the ball-milled process.

The suspensions were poured into metallic molds and then vibrated to eliminate the bubbles in the suspensions. Then, the molds were placed inside a freezing chamber with the temperature of  $-20\ ^\circ\text{C}$  and froze for more than 4 h. The frozen samples were put into the lyophilizer (TIG-A, Zhongke Biologic Co., Ltd, China) for 24 h to remove the ice crystals. The condenser temperature was  $-68\ ^\circ\text{C}$  and the condition of the drying oven was  $60\ ^\circ\text{C}$  with vacuum degree of 4 Pa. Before sintering, the freeze-dried green bodies were heated up in vacuum to  $600\ ^\circ\text{C}$  for 2 h to remove the organic additives. Finally, the samples were divided into three batches, which were sintered respectively at  $2000\ ^\circ\text{C}$ ,  $2050\ ^\circ\text{C}$ , and  $2100\ ^\circ\text{C}$  for 2 h in Ar atmosphere. The obtained porous ceramics were named as 2.5P2000, 2.5P2050, 2.5P2100; 5P2000, 5P2050, 5P2100; 7.5P2000, 7.5P2050, 7.5P2100; and 10P2000, 10P2050, 10P2100 ceramics, respectively.

Phase analysis of sintered porous SiC ceramics was conducted by X-ray diffraction (XRD; D/max 2550 V, Rigaku, Cu K $\alpha$ ,  $\lambda=0.15406\ \text{nm}$ ). Morphologies were observed by scanning electron microscopy (SEM, JSM-6700F, JEOL, Akishima, Japan). The open porosities of sintered SiC ceramics were measured by the Archimedes method. Pore size distributions were characterized by the mercury porosimetry (Model Pore Sizer 9320, Micromeritics, Norcross, GA, USA). Specimens were machined to a dimension of  $3.0 \times 4.0 \times 36.0\ \text{mm}^3$  to test the flexural strength at room temperature via the three-point bending test (Model AUTOGRAPHAG-I, Shimadzu Co. Ltd., Japan) with a support distance of 30.0 mm and a cross-head speed of

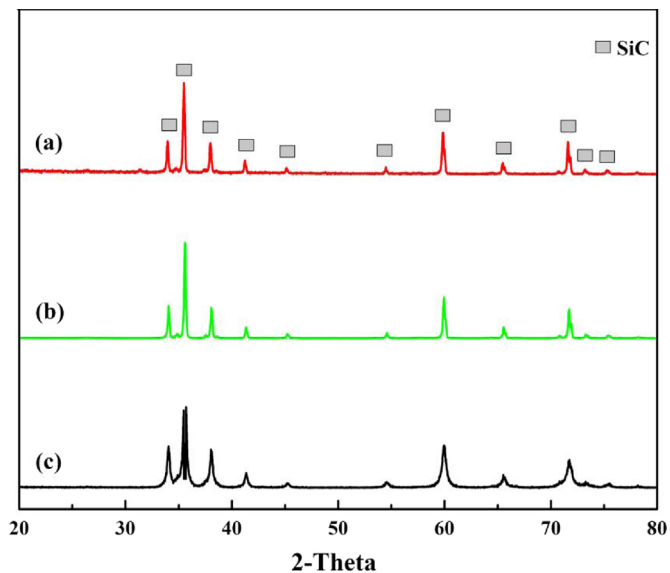
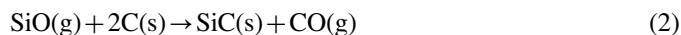
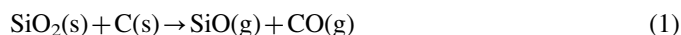


Fig. 1. XRD patterns of (a) porous 2.5P2050 ceramics, (b) porous 10P2050 ceramics, (c) raw SiC powder.

$0.5\ \text{mm}\ \text{min}^{-1}$ . Four samples for each composition were measured to get the flexural strength and open porosity.

## 3. Result and discuss

Fig. 1 presents the XRD patterns of the porous 2.5P2050 and 10P2050 ceramics. The result indicates that the main phase in 2.5P2050 and 10P2050 ceramics is 6H-SiC. The decomposition of PVA produces about 10 wt% residual carbon, [19] the carbon coming from both additives and decomposition of PVA can react with SiO<sub>2</sub> on the surface of SiC particles, as indicated in Formulas (1) and (2). Although the PVA content increases from 2.5 wt% to 10 wt%, no apparent residual carbon was detected in porous ceramics. Thus, the effects of residual carbon on the final properties of porous SiC ceramics are negligible.



### 3.1. Effect of PVA addition on the mechanical properties of SiC

It has reported that PVA introduced as a binder can also facilitate in controlling the ice growth at the same time [20,21]. Zuo et al. have successfully fabricated HAP ceramics and YSZ ceramics with the use of PVA. Ceramics with different pore structures and mechanical properties were obtained through controlling the concentration of PVA in the slurries, while the effects of PVA in these researches varied with the composition of the slurries. The microstructure of 2.5P2050 and 10P2050 ceramics is exhibited in the Fig. 2. The macropores in the ceramics are generated by sublimating of large ice crystals. Large quantities of micropores are observed in the ceramic

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