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Short Communication

Fabrication of porous silicon carbide ceramics with high porosity and high strength

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Abstract

Unique porous SiC ceramics with a honeycomb structure were fabricated by a sintering-decarburization process. In this new process, first a SiC ceramic bonded carbon (SiC/CBC) is sintered in vacuum by spark plasma sintering, and then carbon particles in SiC/CBC are volatized by heating in air at 1000 °C without shrinkage. The honeycomb structure has at least two different sizes of pores; ~20 μm in size resulting from carbon removal; and smaller open pores of 2.1 μm remaining in the sintered SiC shell. The total porosity is around 70% and the bulk density is 0.93 mg/m^3. The bending and compressive strengths are 26 MPa, and 105 MPa, respectively.

Keywords: Porous ceramics; SiC; Porosity; Strength

1. Introduction

Because porous SiC has a low thermal-expansion coefficient, good thermal-shock resistance, excellent mechanical and chemical stability at elevated temperatures it is suitable for such applications as hot-gas or molten-metal filters, catalyst supports, battery electrodes, heat insulators, ion exchangers and others.[1–3] A number of manufacturing processes have been applied to form porous SiC ceramics, including replica techniques, sacrificial template techniques, and reaction techniques.[4–6] Porous SiC requires sufficient strength, controllable porosity, and uniform pore size distribution to enhance or expand its applications.

Recently, we proposed and developed a novel carbon-based ceramic composite, called ceramic bonded carbon (CBC).[7] CBC has a honeycomb microstructure consisting of uniformed carbon particles and a thin ceramic boundary layer. The ceramic network is designed to bond carbon particles, which results in high density, strength, and other functional properties as required.[8,9] Following the CBC concept, a new approach to form porous SiC is proposed, i.e., porous SiC with a honeycomb microstructure consisting of unformed pores and a continuous SiC shell may be obtained by burning out carbon particles of the SiC/CBC. Compared with reported sacrificial template techniques on forming porous ceramics,[5] in this approach SiC is densified by pressure sintering prior to the pore formation. Carbon sacrificial templates are uniformly distributed in the dense SiC matrix. Therefore, the dense SiC shell network provides sufficient strength and the uniform spherical carbon particles ensure a controllable porosity and pore size distribution. Porous SiC parts of complex shape can also be easily fabricated by machining before the decarburization.

In this study, dense SiC/CBC with 30 vol% SiC were decarburized at 600 °C and 1000 °C in air to prepare porous SiC. Phases and microstructure, mechanical properties, and pore size distribution of porous SiC were characterized.

2. Experimental procedure

2.1. Sample preparation

The starting materials included carbon powder made from meso phase pitch carbon by a graphitization step at 2500 °C (Toyo Tanso Co. Ltd.) and Si_3N_4 powder (UBE-10, UBE. Co. Ltd.) that contained 6 wt% Al_2O_3 and 3 wt% Y_2O_3 as sintering

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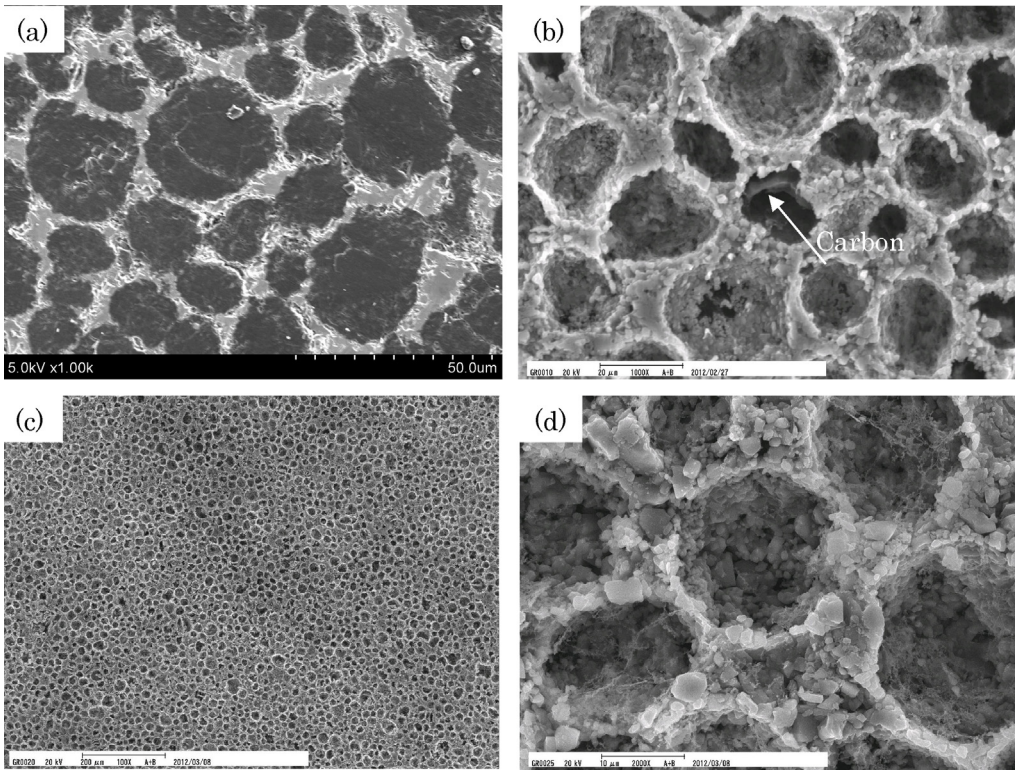

Fig. 1. Microstructure of (a) original SiC/CBC; (b) decarburized at 600 °C for 10 h; (c) decarburized at 1000 °C for 5 h; (d) high magnification of (c).

aids. Both carbon and Si_3N_4 particles have a spherical morphology of approximately 20 μm and 0.5 μm in size, respectively. After densification at 1900 °C for 5 min under 30 MPa by spark plasma sintering in vacuum, Si_3N_4 is converted to SiC by reaction with carbon. The details of the fabrication method are described in our previous report.[8] The microstructure of SiC/CBC is shown in Fig. 1a, in which spherical carbon particles are bonded by thin SiC boundary layers. The bonding is achieved by SiC grains anchoring into carbon particles.

In order to form porous SiC, SiC/CBC (containing 30 vol% SiC) samples were decarburized in air at 600 °C for 10 h or 1000 °C for 5 h. The heating and cooling rate were 10 °C/min.

2.2. Characterization

The bulk density of the porous SiC was calculated from the weight-to-volume ratio of the samples. Microstructure characterization was carried out using a field emission scanning electron microscope (FE-SEM, ERA-8800, Elionix). To characterize compositions of the porous SiC, ground powders were analyzed by X-ray diffraction (XRD, UltimaIV, Rigaku). Rectangular bars of porous SiC, around 3 mm × 2 mm × 20 mm, and 2.2 mm × 2.2 mm × 4.8 mm were used to measure the three-point bending strength and compressive strength, respectively using a Table-Top Universal Tester (EZ-Test Type S, Shimadzu). The speed of the crosshead displacement was 0.5 mm/min during the measurement. The open porosity and pore size distribution were measured using a mercury porosimetry (AutoPore IV 9400, Micromeritics, USA).

3. Results and discussion

3.1. Microstructure and composition

After decarburization, the porous SiC samples retain their size and shape as the original SiC/CBC because the SiC network has already been sintered at 1900 °C by spark plasma sintering (SPS). Fig. 1b shows the microstructure of porous SiC after decarburization at 600 °C for 10 h. Compared with SiC/CBC, decarburization leaves behind spherical pores. The SiC boundary network is converted to a SiC shell network. However, carbon particles are not completely burned out at this phase because of the low temperature and short time. Even at the surface of the sample, carbon particles are still observed. XRD results, as shown in Fig. 2 also show the existence of residual carbon.

In order to completely burn out carbon particles, the SiC/CBC was decarburized at 1000 °C for 5 h in air. As shown in Fig. 2, carbon peaks disappear in the XRD pattern, but weak SiO_2 peaks are observed resulting from oxidation. Because the decarburization rate is related to the sample size, furnace space, and other factors, it is difficult to determine the optimum condition for the decarburization of SiC/CBCs. But based on the above results, we conclude that a pure porous SiC is obtained by decarburization of SiC/CBCs at between 600 °C and 1000 °C after optimizing all parameters.

In addition, because the spherical carbon particles have a uniform size around 20 μm, the replicated pores should have the same uniform size. As shown in Fig. 1c, the isolated pores with a size of 20 μm are uniformly distributed in the SiC continuous matrix. In the higher magnification, SiC shells have a thickness