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# Development of multi-functional NITE-porous SiC for ceramic insulators

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A B S T R A C T

Porous silicon carbide (SiC) ceramics are being considered as functional materials for advanced energy systems due to their low thermal and electrical conductivity, low thermal-expansion coefficient, good thermal-shock resistance, and excellent mechanical and chemical stability at elevated temperature. However, conventional processing routes for SiC are complicated and conventional porous SiC shows poor mechanical and chemical stability at high temperature. Therefore, it is desirable to develop a simple fabrication method. In this study, porous SiC ceramic have been fabricated based on the NITE process, a recently developed processing technique for high performance $SiC_f$/SiC composites. Ceramic porosity was calculated from relative and theoretical density, which was obtained by the rule of mixture. The port shape and size distribution were examined by optical microscopy and scanning electron microscopy. Mechanic properties were evaluated using three-point bend and tensile testing. Thermal conductivity was measured by the laser flash method from room temperature to 900 °C.



## 1. Introduction

Recently, there has been an increasing interest in the applications of porous ceramics as hot-gas or molten-metal filters, catalyst supports, battery electrodes, heat insulators, ion exchangers, gas sensors, and water cleaners. In particular, porous SiC ceramics are considered as functional materials of advanced energy systems, such as perforated containment wall or flow channel inserts (FCIs) for blanket module of fusion reactor, and inner/outer tube of a coated particle type fuel compartment for horizontal flow cooling concept with directly cooling system on Gas-Cooled Fast Reactor, because of their low thermal-expansion coefficient and thermal conductivity, good thermal-shock resistance as well as excellent physical and chemical stability at elevated temperature [1–3].

FCIs made of a SiC fiber reinforced SiC matrix ($SiC_f$/SiC) composite were first proposed by Tillack and Malang [4] as a means for electrical insulation between the flowing liquid metal and the load-carrying channel walls to reduce the magnetohydrodynamic pressure drop in the dual-coolant lead lithium blanket channels of a fusion power reactor. The main attraction of the FCIs is that $SiC_f$/SiC composite has relatively low electrical conductivity, allowing for sufficient reduction of the induced electric currents by decoupling the liquid metal flow from the walls. Another potential advantage of the FCIs is related to low thermal conductivity of the $SiC_f$/SiC composite, which allows for the reduction of heat losses from the breeder and therefore high bulk temperatures at the blanket exit, making the overall thermal efficiency of the blanket higher [5–9].

However, manufacturing method for $SiC_f$/SiC composites is very difficult and complicated. On the other hand, porous SiC is manufactured through a simple procedure than the $SiC_f$/SiC composite. In addition to, porous SiC ceramics has relatively low electrical and thermal conductivity. For these reason, porous SiC ceramics is one of a candidate material as the FCIs in the dual-coolant lead lithium (DCLL) blanket module.

A number of manufacturing approaches have been applied to fabricate porous SiC including polymer pyrolysis [10], oxidation bonding [12], and reaction bonding [11,13–15]. However, their processes are complicated and conventional porous SiC shows insufficient physco-chemical stability under high temperature environment. Therefore, from the view point of safety and stability, it is necessary to develop an uncomplicated manufacturing method and to investigate mechanical and thermal properties of porous SiC ceramics. Therefore, in this study, porous SiC ceramics have been manufactured based on the Nano Infiltration Transient Eutectic process (NITE process), which is developed as a processing technique for $SiC_f$/SiC composite [16]. Furthermore, mechanical and thermal properties of the manufactured NITE-porous SiC ceramics were investigated.

## 2. Experimental procedure

The fabrication process of NITE-porous SiC ceramics has been described elsewhere [17]. Briefly, SiC nano powder (β-SiC, purity > 99.0%, mean particle size 50 nm, HeFei, China) and carbon powder (mean particle size 80 nm, Cancarb, Canada) were used

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as the starting materials. For sintering additives, we used $Al_2O_3$ (purity > 99.99%, mean particle size 0.3 μm, Kojundo Chemical Lab. Co. Ltd., Japan) and $Y_2O_3$ (purity > 99.99%, mean particle size 0.4 μm, Kojundo Chemical Lab. Co. Ltd., Japan). Mixed powders were hot-pressed under a pressure of 20 MPa at 1900 °C for 1 h with a heating rate of 10 °C/min. Sintering atmosphere was argon. And then, porous SiC ceramics could be fabricated by the decarburization process at 700 °C in air. Bulk density of NITE-porous SiC ceramics was measured by the Archimedes method, using distilled water. Porosity was calculated from relative density and theoretical density, which were obtained by the rule of mixture to the volume fraction for the constituents.

Flexural strength of NITE-porous SiC ceramics was measured using three-point bend testing. Rectangular bar 25.0 mm × 4.0 mm × 2.5 mm was prepared by grinding and cutting. The tensile faces of the bars were subsequently polished down to 1 μm diamond polish. The tensile edges were beveled to decrease the effect of edge cracks. All tests were conducted on a conventional screw-driven loading frame (Model 5581, Instron, UK), with a crosshead speed of 0.5 mm/min, using a three-point bending jig of 18 mm support span, at room temperature.

Thermal diffusivities and specific heat capacities were measured by the laser flash method [18] using a thermal analyzer (Ulvac-rico, TC-7000). The laser flash method relies on the generation of a thermal pulse on one face of a thin sample and the recording of the temperature history on its opposite face. The thermal diffusivity can be determined from the time required to reach one-half of the peak temperature in the resulting temperature rise curve on the rear surface. Measurements were performed under argon atmosphere at room temperature to 900 °C. NITE-porous SiC ceramics was cut in other to prepare disk-shaped samples 10 mm in diameter and 2 mm thick. The thermal conductivity of the specimens was then obtained according to the following equation:

$$k = \alpha C \rho, \tag{1}$$

where $k$ is the thermal conductivity, $\alpha$ is the thermal diffusivity, $C$ is the specific heat, and $\rho$ is the density of the specimen.

## 3. Results and discussion

### 3.1. Porosity and microstructure

Porosity of the NITE-porous SiC ceramics is shown in Fig. 1. In this study, target porosities of the NITE-porous SiC ceramics were 30%, 40%, and 50%. As shown in Fig. 1, porosities of the NITE-porous SiC ceramics were 30.2±0.4%, 40.3±0.3%, and 49.7±0.5%, respectively. In other words, porosity of the NITE-porous SiC ceramics could be controlled at less than ±0.5% by the change of amount of carbon particles [17].

Fig. 2 shows the microstructure of the NITE-porous SiC ceramics. Carbon particles could not be observed in all specimens, namely, almost carbon particles were removed by the decarburization process at 700 °C in air. It means that most of the pores in the NITE-porous SiC ceramics were formations of the open pore. Additionally, as shown in Fig. 2, it was confirmed that mean pore size of the NITE-porous SiC ceramics was 0.5 μm. The mean pore size of the NITE-porous SiC ceramics was not changed by the porosity. Accordingly, pore size of the NITE-porous SiC ceramics depends

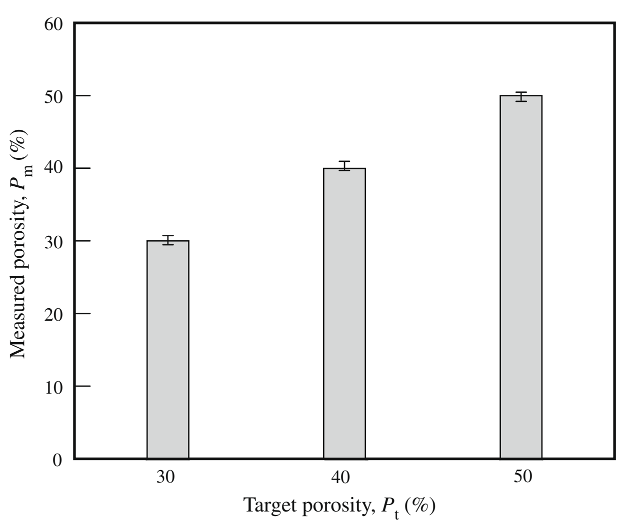


**Fig. 1.** Porosity of NITE-porous SiC ceramics.

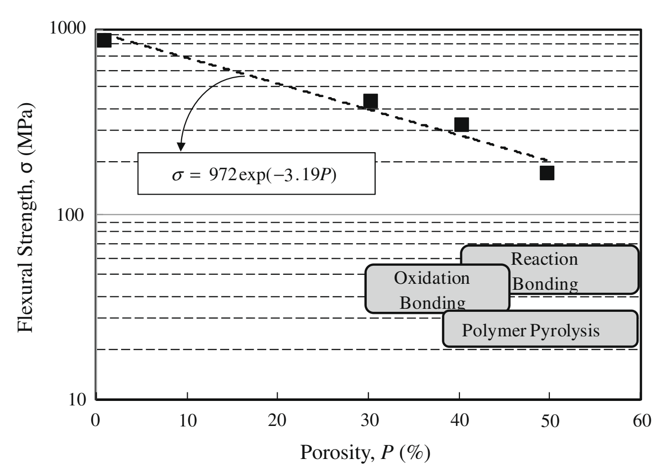


**Fig. 3.** Flexural strength of NITE-porous SiC ceramics.

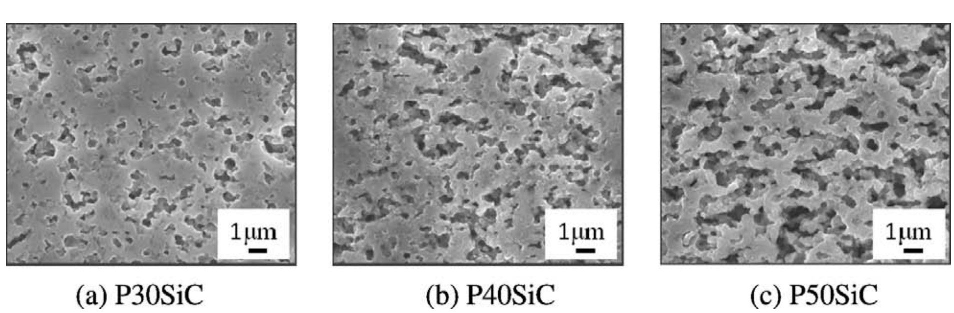


**Fig. 2.** Microstructures of NITE-porous SiC ceramics.

on the size of carbon particles instead of quantity of the carbon particles.

### 3.2. Mechanical properties

Flexural strengths of the NITE-porous SiC ceramics are shown in Fig. 3. Flexural strengths of the NITE-porous SiC ceramics, which has porosity of 30%, 40%, and 50% were about 445±10, 320±10, and 175±5 MPa, respectively. Generally, the mechanical properties of porous ceramics depend strongly on porosity. The mechanical properties–porosity dependence can be approximated by an exponential equation as follows:

$$\sigma = \sigma_0 \exp(-bP), \tag{2}$$

where, $\sigma_0$ is the flexural strength of a nonporous structure, $\sigma$ is the flexural strength of the porous structure at a porosity $P$, and $b$ is a constant that is dependent on the pore shape. The values of $b$ were reported to be 1.4 for cylindrical pores, 3 for spherical pores, and 5 for solid spheres in cubic stacking [19]. The fit of this equation to the results in flexural strength gave $\sigma_0 = 972$ MPa and $b = 3.19$. The flexural strength–porosity behavior of the NITE-porous SiC ceramics can be described by the following equation:

$$\sigma = 972 \exp(-3.19P). \tag{3}$$

As discussed above, main shape of the pores in the NITE-porous SiC ceramics can be viewed as a spherical pore. Consequently, the NITE-porous SiC ceramics exhibited a substantially high strength in comparison with other conventional porous SiC ceramics, due to its robust microstructure consisted of spherical pores [17].

### 3.3. Thermal properties

Figs. 4 and 5 show the mean specific heat and the thermal diffusivity of the NITE-porous SiC ceramics at elevated temperature, respectively. Mean specific heat of the NITE-porous SiC ceramics, as shown in Fig. 4, was not changed by porosity. It means that carbon particles were completely removed by the decarburization process. In other words, all NITE-porous SiC ceramics have been constructed from same component. As shown in Fig. 5, thermal diffusivity of the NITE-porous SiC ceramics decreased with increasing porosity and temperature. From the decreasing thermal diffusivity of the NITE-porous SiC ceramics, low thermal conductivity at elevated temperature could be expected. Fig. 6 shows the variation of thermal conductivities by the measurement temperature of the NITE-porous SiC ceramics with results of $SiC_f/SiC$ composites included for comparison [20]. $SiC_f/SiC$ composite is dominant candidate material for SiC FCIs of DCLL blankets, because of their low thermal and electrical conductivity. However, thermal conductivity of the NITE-porous SiC ceramics with 40% and 50% porosity is lower than the value of $SiC_f/SiC$ composite. As a matter of course, thermal conductivity of the NITE-porous SiC ceramics with 50% porosity was below 4 W/m/K from 600 °C to 800 °C. Based on these preliminary results, it appears that NITE-porous SiC ceramics may be attractive materials for advanced energy systems.

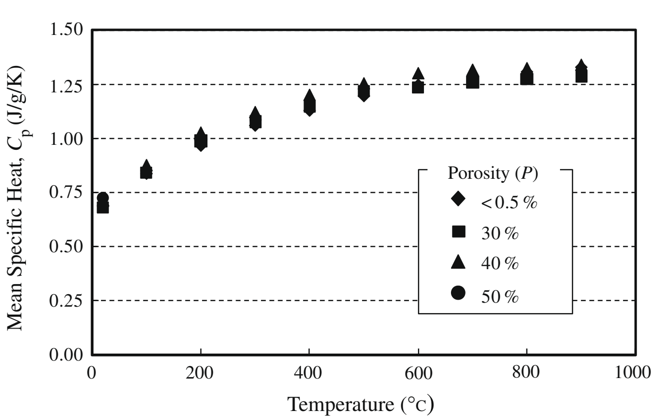


**Fig. 4.** Mean specific heat of NITE-porous SiC ceramics.

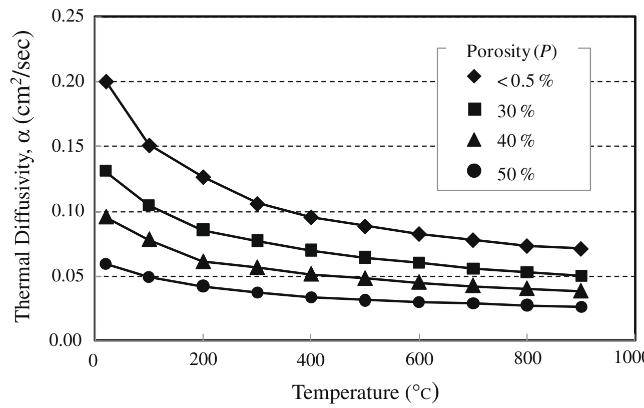


**Fig. 5.** Thermal diffusivity of NITE-porous SiC ceramics.

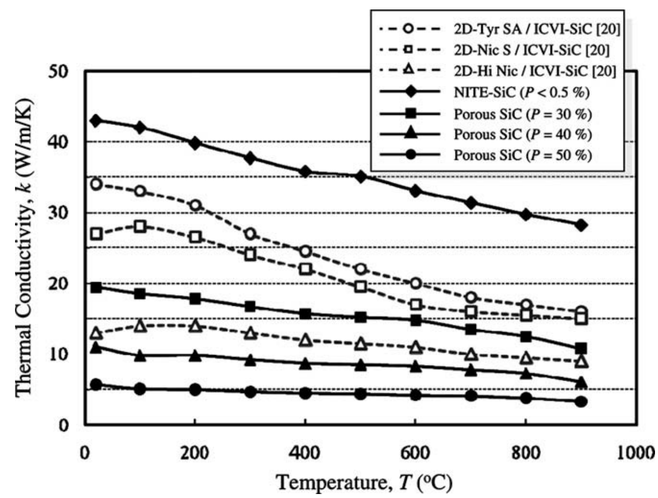


**Fig. 6.** Thermal conductivity of NITE-porous SiC at elevated temperature.

## 4. Conclusion

Porous SiC ceramics have been manufactured based on the NITE process. Porosity of the NITE-porous SiC ceramics could be controlled with a high degree of accuracy by the change of amount of carbon particles. The NITE-porous SiC ceramics exhibited a substantially high strength in comparison with other conventional porous SiC ceramics, due to its robust microstructure consisted of spherical pores. Also, the NITE-porous SiC ceramics had low thermal conductivity from room temperature to 900 °C. Therefore, from the good mechanical and thermal insulation properties, NITE-porous SiC ceramics could be expected as high performance multi-functional materials for advanced energy systems.

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