Performance-properties and fabrication of the reaction sintered Si/SiC honeycomb materials for solar absorber application

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Reaction sintered Si/SiC composite was fabricated to apply to a solar absorber and the fabricated material was evaluated on its basic properties and performance. SiC and carbon black were mixed to shape a honeycomb with a multi-channel via vacuum extrusion process. The Si/SiC honeycomb material was also fabricated with less than 5% of pores by molten silicon infiltration in vacuum. The sintered density and porosity of the fabricated material, and the 3-point bending strength at room temperature and high temperature (1100–1300°C) were measured and the thermal conductivity at room temperature and 1100°C were also analyzed. In addition, the fabricated honeycomb material was modulized and installed to a solar absorber system to measure the outlet air temperature and thermal efficiency and evaluate the performance of honeycomb material as a solar absorber material.

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1. Introduction

Silicon carbide is a candidate material that can be utilized in various fields such as high temperature, aerospace, energy, electronic, nuclear power and transportation.¹⁾ Ever since its development by P. Popper, reaction-bonded SiC, RBSC, has been widely used in fields that require wear and corrosion resistance.^{2),3)} RBSC ceramics are produced through the penetration of SiC/C or carbon preforms as molten silicon. The carbon and molten silicon reaction leads to the formation of β -SiC and the interconnection of the particles of the starting material, SiC, to form a continuous ceramic structure.^{4),5)} Unlike other structural ceramics, RBSC is advantageous in fabricating near-net shape sintered objects because it maintains the shape of preforms due to very little dimensional changes and an insignificant number of pores after the final densification sintering. In addition, the fabrication of a quality RBSC product is possible without using high purity and fine particle SiC powder, and the short process time lowers the production cost for commercialization.^{6)–8)}

SiC-based ceramics are applicable to the use of solar energy and high temperature applications because they have especially outstanding thermal characteristics. In addition, the outstanding thermal conductivity, SiC, is inherently of black color, which maximizes the absorbance by effectively heating up the gas within the channels when it is made into a honeycomb shape for solar concentration.^{9)–11)} Thin wall multi channeled ceramic honeycombs have been commercially considered as candidate materials for various industrial applications such as automotive catalysts, catalytic combustion and hot gas cleanup filters. Advantages of

the ceramic honeycombs include thin wall, geometrically high specific surface area. Therefore it indicates good gas-solid contact, control of high gas flow rates combined with low pressure drop and excellent mass transfer performance.

Volumetric solar receivers (or solar absorbers) with high porosity honeycombs and/or foams have been used in the solar tower technology for the conversion of concentrated solar radiation into heat.^{12)–14)} This technology has been developed during the past 20 years, a number of large scale tests have been carried out.^{15)–19)}

Especially, Agrafiotis et al.¹³⁾ were evaluated with variety of recrystallized SiC materials with respect to pore structure and thermomechanical properties in the “as-manufactured” state as well as after prolonged operation as solar thermal collectors under solar irradiation. The goal was to understand the phenomena that take place under solar irradiation and increase the lifetime of exposed receiver elements. In addition, an alternative material, reaction-bonded SiC known to exhibit superior mechanical properties and oxidation resistance was also explored to meet the demands for prolonged receiver lifetime.

Generally, recrystallized SiC, with a connected pore structure and excellent thermomechanical properties, and reaction sintered SiC, impregnated with metallic silicon with excellent mechanical properties and oxidation resistance, are highlighted as materials for the volumetric solar receiver.

Therefore, this research utilized SiC as a raw material and shaped honeycomb material through extrusion. Then, densified volumetric solar receiver material with multi-channel was fabricated by LSI (Liquid Silicon Infiltration) process. And its mechanical and thermal properties were evaluated and performance of the honeycomb material was performed in a volumetric solar receiver system.

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2. Experimental

2.1 Raw materials and compositions

The materials used for the fabrication of the honeycomb for a volumetric solar receiver were SiC, the raw material for the supporting layer, in average diameters of 44, 85, and 100 μm (98%, China Abrasives Corp., China) mixed in a unimodal or bimodal composition. The powder, added with SiC powder, was carbon black with an average diameter of 70 nm (99.9%, HIBLACK L-30, LG Chem., Korea). In addition, 1–2 mm (98%, China Abrasives Corp., China) of granule metallic silicon powder was used for the LSI process.

A MC (methylcellulose)-based binder, YB-132A (Yuken Industry Co., Japan), was used for the extrusion molding in order to shape the honeycomb, and **Table 1** lists the combination ratios.

2.2 Preparation of honeycomb preforms

The raw materials listed in Table 1, SiC and carbon black powders were primarily dry blended in a Σ -blade kneader for the shaping of the honeycomb material. Organic binder and water were added to the primarily mixed raw materials and sealed after secondary wet mix. Then, the material was aged for at least 24 h and used as the raw material for extrusion. Honeycomb preforms were produced as the raw materials mixed in the vacuum extrusion machine passed through a mold manufactured to produce a 40 mm $\times$ 40 mm area and a channel of 200 CPSI (Channels Per Square Inch).

The shaped honeycomb was completely dried of moisture in a dryer that can adjust the temperature to prevent distortion or

cracks. Then, the shaped honeycomb was fabricated in a vacuum furnace by both a reaction sintering process by LSI process. The LSI sintering was conducted by increasing the temperature from room temperature to 600°C at 1°C/min to start the debinding process of organic binders within the preforms. Then, the temperature was increased upto 1650°C at 5°C/min and the temperature was maintained for 30 min in a vacuum furnace that maintains 10^{-1} torr. As listed in Table 1, the preform was sintered on silicon powder, which was of equal amount as the mass of the preform, in a BN (Boron Nitride) coated graphite crucible.

2.3 Properties and performance evaluation

It is difficult to evaluate the characteristics of the material for the honeycomb type sintered materials from extrusion because the thickness of the bulkheads forming the channels was only 0.4 mm. Thus specimens were extruded in a 5 mm W $\times$ 3 mm H bar shape with the combination listed in Table 1. The specimen was cut in 45 mm length and sintered by LSI process. Its 3-point bending strength in room and high temperatures was evaluated, and the edges of every specimen for measuring the bending strength were chamfered prior to the measurements. The strength at room temperature was measured by UTM (H10KS, Hounsfield, UK) at 0.1 mm/min cross head speed and the strength at high temperature was measured by UTM (INSTRON 4505, U.S.) in vacuum atmosphere at 15°C/min for 1000°C, and at 0.5 mm/min in 1150 and 1300°C at 0.5 mm/min cross head speed. The sintered density and porosity were measured from fractured specimens after measuring the bending strength by Archimedes method. The microstructures of the polished surface and fracture surface from the measuring bending strength were observed with an optical microscope (EPIPHOT 300, NIKON, Japan) and a field scanning electron microscope (FE-SEM, S-4700, Hitachi, Japan), respectively. And the thermal conductivity was measured with a Laser Flash Apparatus (Netzsch, LFA-475, Germany) at room temperature and high temperature. In the measurement of the thermal conductivity of a material based on the laser flash method, the thermal diffusivity (mm^2/s) and thermal capacity meaning specific heats ($\text{J/g}\cdot\text{K}$) are measured and their values are multiplied with density (g/cm^3) to calculate thermal conductivity ($\text{W}/\text{m}\cdot\text{K}$).

The performance test of Si/SiC honeycomb material in solar receiver system carried out in the present work. The volumetric solar receiver module was installed in a solar furnace system to

Table 1. Raw materials and compositions for SiC honeycomb material preparation by vacuum extrusion process

Samples	Material	Particle size (μm)	Content (wt %)
S325-C20	SiC	44	80
S325/180-C20	SiC	44 + 85	80
S325/150-C20	SiC	44 + 100	80
	Carbon black	0.07	20
	Silicon	1000–2000	100
	Binder	—	5
	Water	—	120

Fig. 1. Schematic diagram of the ambient pressure solar receiver apparatus.

measure the exit temperature of air and efficiency as shown in Fig. 1. The outer and internal diameters of the parabolic reflector are 4.5 and 0.75 m, respectively, and the focal distance is 4.98 m. The characteristics of high temperature concentration facilities are obtained by heat flux measurement methods using a radiometer and CCD camera, and the system used in this experiment has the capacity of a 3,500 sun concentration ratio and 40 kW t.²⁰⁾ The input power (kJ) in front of the Si/SiC honeycomb material can be obtained when the solar furnace concentrates on the light and is utilized with the heat flux measurement results. The input power value varies with respect to the direct normal insulation and reflectivity at the moment of the experiment. In this work, the performance evaluation of honeycomb material was conducted in the range of 6.7–10.4 kW. The efficiency of the honeycomb material can be computed by measuring the air mass (kg) and temperature at the exit and computing the energy obtained by the air.

3. Results and discussion

3.1 Sintered density and porosity

Figure 2 illustrates the sintered density and porosity of the sintered (S325/180-C20, S325/150-C20) composite body of the mixture of 44 μm in unimodal SiC powder composition. Two SiC powders with different particle sizes were also mixed in bimodal. S325-C20 was composed of 44 μm SiC and 20 wt% carbon black. S325/180-C20 was composed of 44 and 85 μm SiC powders in 1:1 weight ratio with 20 wt% carbon black, and S325/150-C20 was composed of 44 and 100 μm SiC powders in 1:1 weight ratio with 20 wt% carbon black composition.

The sintered densities of S325-C20 and S325/180-C20 compositions were measured to be 2.97 g/cm³, and that of the S325/150-C20 composition was 2.94 g/cm³. The porosities of S325-C20, S325/180-C20, and S325/150-C20 were 0.75, 0.57, and 0.84%, respectively.

As shown in Fig. 2, in the three composition specimens of the sintered body, the porosity is showing some difference from, but it is reveal that dense reaction-bonded SiC is fabricated with nearly pore free bodies, because of the standard deviation value at each 0.38, 0.48 and 0.61 less than 1% porosity range. Therefore, these deviation results seems that a bimodal particle size composition relative to a unimodal particle composition is relatively improves the packing density during pressing of the green bodies in accordance with a some fine closed pore randomly distributed in interior structure.

Fig. 2. Sintered density and porosity of the SiC honeycomb materials by LSI process.

3.2 Room and high temperature flexural strength

Figure 3 illustrates the 3-point bending strength measured in room temperature. The strengths were in the order of S325-C20 > S325/180-C20 > S325/150-C20 and the values were 272, 241, 203 MPa, respectively. Such result is in agreement with the porosity and sintered density measurement results, and the reaction sintered monolithic SiC sintered bodies conventionally produced from silicon melt infiltration had characteristics of porosities of less than 2%. The fabrication of the pore-free sintered body, with less than 1% of porosity, has been verified through this research, and compositions with a high sintered density yielded high bending strength.

Figure 4 illustrates the bending strengths of three composite sintered specimens measured in high temperature. Just like room temperature strength over every temperature range, high temperature strengths were in the order of S325-C20 > S325/180-C20 > S325/150-C20.

Considering the results, unimodal SiC compositions have relatively higher high temperature strengths than the high temperature strengths of the bimodal SiC compositions. Using the high temperature strength at 1300°C as the standard, the strength illustrated the trend of increasing strength relative to the room temperature strength along with the trend of decreasing strength with increasing measuring temperature. The decreasing high

Fig. 3. Room temperature 3-point flexural strength of the SiC honeycomb materials by LSI process.

Fig. 4. High temperature 3-point flexural strength of the SiC honeycomb materials by LSI process.

temperature strength with an increasing measuring temperature was caused by the softening of the solid metallic silicon in the residual pores as free Si around the molten temperature of 1412°C.

3.3 Thermal conductivity

For dense SiC honeycomb material sintered bodies, the thermal conductivities of specimens with S325-C20 composition which indicated most high strength value were representatively measured at room temperature and 1000°C, and the results are listed in **Table 2**. For the measurements of thermal conductivities, specimens with a S325-C20 composition were extruded in a plate shape, and the sintered bodies with the methods mentioned earlier were processed to be 12 mm OD × 1 mm T circular specimens. The thermal conductivities were also measured after surface polishing.

The thermal conductivities were measured to be 118 and 35 W/mK at room temperature and 1000°C, respectively. Both thermal conductivity and thermal diffusivity at room temperature are lower than those of 1000°C. However, the heat capacity is higher than that of 1000°C. Those are good agreement with the results of porous SiC ceramics fabricated by pressureless sintering.²¹⁾ Thermal conductivity at higher temperatures than 1000°C is more important than at room temperature value for the material to be used in a volumetric solar receiver. This result proves that this material has an outstanding thermal conductivity characteristic compared to other materials⁹⁾ reviewed as candidate materials for the volumetric solar receiver.

Thermal conductivity, K , is an intrinsic property that indicates the heat transfer capacity of a material. Unlike metals, in which heat is transferred by electrons, most of the heat is transferred primarily by phonons in ceramics. A phonon is a unit quantum of vibration and the total number of phonons in a system is directly related to the temperature of a crystal, which is the system. Therefore, when the temperature increases, a vibration of object materials increases and its number of phonon also increases.

Phonon-Phonon interaction plays an essential role in thermal conduction in ceramics, and in a perfect crystal, the movement of a phonon is easily dispersed by other phonons as the temperature increases. However, in real dielectric solids, there are imperfections such as pores, impurities, crystal boundaries and dislocations.

Therefore, the mean free path of a phonon decreases rapidly and it is reported to be expressed in a Matthiessen Rule equation.²²⁾

$$\frac{1}{\lambda} = \frac{1}{\lambda_{\text{defect}}} + \frac{1}{\lambda_{\text{boundary}}} + \frac{1}{\lambda_{\text{phonon}}} \quad (1)$$

Therefore, the thermal conductivity of a dielectric material is given by the below equation. Here, ρ , C_p and v represent the bulk density (g/cm³), heat capacity (J/g·K), and elastic wave velocity (cm/s), respectively.

Table 2. Thermal conductivity of the reaction sintered SiC honeycomb materials with S325-C20 composition at R.T. and 1100°C under air atmosphere

Temperature (°C)	Diffusivity (mm ² /s)	Specific Heat (C_p , J/g·K)	Density (g/cm ³)	Conductivity (W/m·K)
25.0	54.6	0.732	2.94	117.6
(Std. Dev.)	0.11	—	—	0.24
1100	9.9	1.194	2.94	34.6
(Std. Dev.)	0.01	—	—	0.03

$$K = \frac{\rho \lambda v C_p}{3} \quad (2)$$

From the equation, the thermal conductivity of the material is expressed in the below equation, and here α is the thermal diffusivity of a solid.

$$K = \rho C_p \alpha \quad (3)$$

In order to obtain a high thermal conductivity value, it is essential that fine carbon particles and infiltrated molten silicon added to the sintered bodies as starting materials maximize the formation of silicon carbide (SiC) particles through the $Si_{\text{liquid}} + C_{\text{solid}} \rightarrow SiC_{\text{solid}}$ reaction, and increase the sintered density as free Si fills up the excess voids.

In addition, the specific heat of Si (0.79 J/g·°C in R.T.) is higher than the specific heat of SiC (~0.67 J/g·°C in R.T.). Therefore, increasing free Si content is advantageous to improving the thermal conductivity. However, as the result in Fig. 3 illustrates, in a high temperature above 1300°C, the problem of decreasing high temperature strength arises from high free Si content. Therefore, in order to apply reaction-bonded SiC honeycombs as solar absorber materials, it is revealed that free Si content of sintered bodies should be minimized and thus sintered density increase by increasing for carbon contents which must be react to melted Si in the green body.

3.4 Microstructure

Figure 5 illustrates the picture of the microstructure of the polished surface of a sintered body with a S325-C20 composition, which yielded the highest sintered density and bending strength, viewed by an optical microscope, and the picture of the microstructure of the fractured surface viewed by a FE-SEM after measuring the bending strength at room temperature. The picture of the polished surface in Fig. 5(a) illustrates the typical

Fig. 5. (a) Optical micrograph of polished surface and (b) FE-SEM images of fracture surface of the reaction sintered SiC honeycomb materials with S325-C20 composition.

microstructure of a Si/SiC complex fabricated by liquid phase reaction sintering, which does not show any silicon islands, pools, and rivers. The large grey particles (solid white arrows) are the SiC particles added as the starting materials and the smaller grey particles (dotted white arrows) are SiC particles fabricated by the previously mentioned $Si_{liquid} + C_{solid} \rightarrow SiC_{solid}$ reaction. Through this process, the newly generated small SiC particles are dissolved into free Si liquid (solid black arrows) and reprecipitated around the original SiC particles for combination.

On the other hand, Fig. 5(b) illustrates the microstructure of the specimen fractured from the bending strength measurement. The microstructure shows the trend of coexisting transgranular fracture and intergranular fracture (solid arrow). In addition, the striation mark (dotted arrow) along the cleavage plane of the free Si shows that the result is in agreement with the previous studies.²³⁾

Also, as shown in Fig. 6, it can confirm that closed pores are locally distributed in the interior fracture surface of all sintered bodies. In particular, the S325/180-C20 and S325/150-C20 sintered bodies reveal that large cavity defects randomly existed connecting to small pores which does not infiltrated with free Si during reaction sintering process. Thus, it was found that the difference in the flexural strength in spite of similar sintered

Fig. 6. Fracture surface micrographs of the reaction sintered SiC honeycomb materials: (a) S325-C20, (b) S325/180-C20 and (c) S325/150-C20, respectively.

density and porosity of the three specimens caused by these large closed cavity defects existing interior microstructures.

3.5 Performance test of Si/SiC honeycomb material in solar receiver system

Figure 7 shows 120 mm W × 120 mm L × 100 mm H volumetric solar receiver module which stacked and bonded the 40 mm W × 40 mm L × 100 mm H Si/SiC honeycomb unit cell in order to evaluate the thermal transfer performance of reaction sintered Si/SiC honeycomb material.

Figures 8(a) and 8(b) illustrates the average air exit temperature and thermal efficiency measured with respect to the function of air mass flow versus input power with respect to the

Fig. 7. Volumetric solar receiver module fabricated by extrusion and LSI process.

Fig. 8. Comparison of the (a) air inlet temperature and (b) thermal efficiency results for the Si/SiC honeycomb absorber materials under different operational conditions.

hexagonal and rectangular shapes of the unit cell of the honeycomb material. Such method is suitable for comparing the performances of Si/SiC honeycomb materials because the air exit temperature increases proportionally, while the radiation loss increases and efficiency decreases with the increasing temperature. In order to compare the performance of the Si/SiC honeycomb material developed by the present work, the data announced by Pitz-Paal of Germany and SiC honeycomb material measurements of DPF (diesel particulate filter) for vehicles with a porosity lowered to 25% were also presented.²⁴⁾

As Fig. 8 illustrates, SiC material for a low porosity DPF was revealed to be unsuitable for the volumetric solar receiver from the perspective of exit temperature and efficiency. On the other hand, Si/SiC honeycomb material developed by this present work yielded a relatively superior air exit temperature and thermal efficiency. Of the honeycombs this research developed, the use of receiver material with a hexagonal channel structure, the reduced loss of pressure was measured. Effective convection heat transfer within the hexagonal channel structure seems to have compensated for the performance loss that derives from low cell density.

KIER-hexagonal and KIER-square honeycombs in here are prototype solar absorber materials fabricated by S325-C20 composition, and Low-porosity DPF sample is manufactures in domestic company (K. Ltd.) for DPF (Diesel Particulate Filter) materials. This commercialized DPF material was manufactured by ambient sintering at 1400°C using clay contained oxide additives, and its mechanical and thermal properties of the this DPF materials are sintered density 1.8 g/cm³, porosity ~50%, three point-flexural strength (R.T.) 15 ± 2 MPa, thermal conductivity 12 ± 3 W/m·K, respectively. Therefore, because properties of the SiC honeycomb material for DPF is significantly lower than that of reaction-bonded SiC materials, the use of solar absorber materials is considered inappropriate.

In addition, when it is compared with the SiC honeycomb (NoTox L6, Denmark) volumetric solar receiver material by Pitz-Paal in the range of 1300–1400 kJ/kg, the material developed by this research with an exit air temperature and efficiency of 840–860°C and 66–69% was superior to the NoTox SiC, which has the exit air temperature and efficiency of 850°C and 65%.

Figure 9 illustrates the emissivity (ε) spectrum measurement results on Si/SiC honeycomb material. The emissivity was measured with two light sources, deuterium lamp and tungsten lamp, using a UV/VIS/NIR spectrophotometer (Lambda 1050, PerkinElmer, US) in the wavelength region of 0.18–3.3 μ m. Due to the characteristics of the main component, SiC, the trend

Fig. 9. Emissivity of the Si/SiC honeycomb absorber materials in 0.18–3.3 μ m wave length ranges.

of overall high emissivity increasing with the wavelength was clearly exhibited. The value of approximately 0.85 was obtained from the result, and since the emissivity of Si is generally lower than the emissivity of SiC, lowering the free Si content in the reaction sintered Si/SiC sintered body must be an effective method to increase the emissivity.

4. Conclusions

(1) Using SiC as the starting material in the vacuum extrusion to shape a honeycomb with the area of 400 mm × 40 mm L and 200 CPSI unit cell, a reaction sintered Si/SiC composition honeycomb for a volumetric solar receiver was fabricated from metallic silicon melt infiltration.

(2) When the unimodal and bimodal SiC particles were used as the starting materials, high sintered density and low porosity were the result from the use of unimodal SiC particles, and room temperature and high temperature also yielded the same result.

(3) Compared to other materials reviewed as the candidate materials for the volumetric solar receiver, the developed material yielded excellent thermal conductivity characteristics.

(4) The microstructure of the fractured specimen of the Si/SiC honeycomb material showed a trend of coexisting transgranular and intergranular fracture (solid arrow). In addition, the striation mark from the cleavage plane of free Si was also observed.

(5) The performance evaluation of solar thermal conductivity of the developed Si/SiC honeycomb material yielded outstanding performances of average exit air temperature and efficiency, 840–860°C and 66–69%, respectively.

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