Progress in Nuclear Science and Technology Volume 7 (2025) pp. 221-227

ARTICLE

Effects of sintering atmosphere and form of Al and B addition on thermal and electrical properties of silicon carbide ceramics

Ying Chung^{a,*}, Anna V. Gubarevich^b and Katsumi Yoshida^b

^a Department of Materials Science and Engineering, School of Materials and Chemical Technology, Tokyo Institute of Technology, 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8550, Japan; ^b Laboratory for Zero-Carbon Energy, Institute of Innovative Research, Tokyo Institute of Technology, 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8550, Japan

The effects of sintering atmosphere and additive form of aluminum (Al)- and boron (B)- added silicon carbide (SiC) ceramics were investigated. The sintering atmosphere was found to affect both thermal conductivity and electrical properties of Al- and B-added SiC ceramics. Al-added SiC ceramics sintered in Ar possessed lower thermal conductivity and lower electrical resistivity, while B-added samples possessed the opposite. All SiC ceramics sintered in N₂ possessed lower thermal conductivity, but the electrical resistivity of Al-added samples is higher and that of B-added ones was lower. The formation of secondary phases containing N with these two additives and the formation of solid solution of N into SiC in the presence of Al and B are considered to determine how the results of thermal conductivity and electrical resistivity would behave. The different sinterability brought by different additive form is thought to affect these properties of Al- and B-added SiC ceramics.

Keywords: silicon carbide; thermal condutivity; electrical resistivity; sintering atmosphere; addive forms

1. Introduction

Thermoelectric materials that are capable to generate electrical energy from thermal energy based on the Seebeck effect are one of the key technologies attracting attention to decrease the dependency of fossil fuel energy in order to achieve a carbon neutral society. One of the focused applications of thermoelectric materials is collecting waste heat generated from conventional power plants, factories and other industries to improve the energy efficiency. Major thermoelectric materials developed so far mostly contain heavy metals such as bismuth (Bi), tellurium (Te), lead (Pb), tin (Sn) [1]. Therefore, it is difficult for these materials to be applied under high temperature environment due to their low melting point and poor oxidation resistance at high temperature. In order to overcome this obstacle, alternative materials that are low cost, environmentally friendly with stability should be considered.

Silicon carbide (SiC) ceramics have been considered as a potential candidate of high temperature thermoelectric material due to its semiconductor properties, superior thermal stability, high chemical stability and high strength at high temperatures. SiC is expected to be serve as a thermoelectric material to collect waste heat ranging from 500 °C to 1000 °C, and those high temperature waste heat mostly comes from iron and steel industries [2]. In addition, for industrial waste heat, over 40% is above 650 °C [3]. However, SiC is a semiconductor with high thermal conductivity, which makes its figure of merits (ZT), i.e. the efficiency of thermal energy to electrical energy conversion, becomes naturally low. Therefore, developing SiC ceramics with low thermal conductivity and high electrical conductivity is a critical issue for high temperature thermoelectric materials. Nevertheless, the tradeoff effect of low thermal conductivity may cause low electrical conductivity. Fabricating SiC into porous bodies is one of the common approaches to lower thermal conductivity [4], and sintering additives are often used for the fabrication of SiC ceramics. In addition to pores, additives and sintering atmosphere would also affect the thermal and electrical properties of SiC ceramics.

In both porous SiC ceramics with boron nitride (BN) [5] and with boron carbide (B₄C) [6], the electrical resistivity was found to be strongly affected by the sintering atmosphere, and the thermal conductivity was more dependent on the porosity and necking area of SiC grains. Nevertheless, the effect of sintering atmosphere on the electrical properties of porous SiC ceramics with single boron (B) addition or other additives such as aluminum (Al) has not been investigated yet. Besides, the electrical resistivity difference between B₄C- and BN-added porous SiC significantly large although their porosity was similar. Therefore, the effect of additive form is also worth more investigation. In our previous work, by

^{*}Corresponding author. E-mail: chung.y.aa@m.titech.ac.jp

analyzing the occupancy of additive atoms in SiC lattice, solid solution is found to dominate the thermal conductivity of Al- or B-added porous SiC ceramics [7]. However, whether solid solution would also dominate electrical resistivity and the effect of sintering atmosphere on thermal conductivity have not been clarified yet. This study is considered as a fundamental research for thermoelectric properties of SiC ceramics, and the effects of sintering atmosphere and additive form on the thermal and electrical properties of SiC ceramics with Al or B addition were investigated and discussed.

2. Experimental procedure

Beta-SiC powder (BF-17, average particle size: 0.5 µm, Höganäs GmbH, Germany) was used as the starting material. Two different kinds of additives as Al and B sources were used; pure aluminum (99.9%, Kojundo Chemical Laboratory Co., Ltd., Japan) and aluminum nitrate ((Al(NO₃)₃ · 9H₂O), Nacalai Tesque, Ins., Japan), amorphous boron powder (Rare Metallic Co., Ltd., Japan) and boron carbide (B₄C, HS, Höganäs GmbH, Germany), respectively. Two kinds of composition of Al or B source, 0.5 wt% and 5.0 wt%, were added to β -SiC powder. The mixed powder was formed into compacts at uniaxial presssure of 17 MPa, followed by cold isostatic pressing (CIP) of 200 MPa. The compacts were pressurelesssintered in a high temperature furnace (Hi-Multi 5000, Fujidempa Kogyo Co., Ltd., Japan) with a heating rate of 30 °C/min at 2180 °C under argon (Ar) or nitrogen (N₂) flow for 1 hour. Sample codes were given according to the composition and sintered atmosphere as listed in Table 1.

Bulk density (ρ) and open porosity of the samples were measured by the Archimedes' method, and the closed porosity was calculated based on the theoretical density of SiC (3.21 g/cm³ [8]). Their microstructure was observed with a field emission scanning electron microscope (FE-SEM, S-4800, Hitachi High-Tech Co., Japan). Crystalline phases of the samples were identified by powder X-ray diffraction (XRD) analysis (Aeris Research edition, Malvern Panalytical, the Netherlands; Cu-K α). Quantitative analysis of SiC polytypes in the samples and occupancies of additive atoms in SiC lattice was done by Rietveld method on HighScore Plus (Ver. 4.7a, Malvern Panalytical). The thermal diffusivity (α) and heat capacity (C_p) measurements were conducted with a laser flash analyzer (LFA457 MicroFlash, NETZSCH GmbH, Germany) from room temperature to 1000 °C under Ar flow (200 ml/min). The measurements were carried out at least three times every 100 °C from room temperature to 1000 °C, and Pyroceram 9606 (NETZSCH GmbH, Germany) was used as the reference material for C_p measurement. The thermal conductivity (κ) was calculated by the following equation;

$$\kappa = \alpha C_p \rho \tag{1}$$

The electrical resistance (*R*) was measured using the van der Pauw method [9] as shown in Fig. 1, and the electrical resistivity (ρ) was then determined by the following equation;

$$\rho = \frac{\pi d}{\ln 2} \cdot \frac{R_{34,12} + R_{13,24}}{2} \cdot \frac{R_{34,12}}{R_{13,24}} \tag{2}$$

where *d* is the thickness of the sample, $R_{34,12}$ is the resistance between point 1 and 2 that obtained from applying current I_{34} and measuring voltage V_{12} of the sample, and $R_{13,24}$ is the resistance between point 2 and 4 that obtained from applying current I_{13} and measuring voltage V_{24} of the sample (**Figure 1**). To be noted, the last term of Eq. (2), $R_{34,12}/R_{13,24}$, will only be taken into consideration when the sample is anisotropic, which can be ignored here.

3. Results and discussion

3.1. Density and porosity

The relative density of each sample is shown in **Figure 2**. The relative density of Al-added samples increased with increasing the concentration of Al and that of B-added



Figure 1. Schematic illustration of van der Pauw method for electrical resistivity evaluation.

Table 1. Sample code, composition and sintering atmosphere of Al- or B-added SiC ceramics.

Sample code		Sample Composition (wt%) and sintering atmosphere
A-SC05Al-m	A-SC05Al-n	99.5 % β-SiC + 0.5 % pure Al (-m), 99.5 % β-SiC + 0.5 % Al nitrate (-n), Ar
A-SC50Al-m	A-SC50Al-n	99.5 % β-SiC + 5.0 % pure Al (-m), 99.5 % β-SiC + 5.0 % Al nitrate (-n), Ar
A-SC05B	A-SC05BC	99.5 % β -SiC + 0.5 % B, 99.5 % β -SiC + 0.5 % B ₄ C, Ar
A-SC50B	A-SC50BC	99.5 % β -SiC + 5.0 % B, 99.5 % β -SiC + 5.0 % B ₄ C, Ar
N-SC05Al-m		99.5 % β-SiC + 0.5 % pure Al, N ₂
N-SC50Al-m		99.5 % β-SiC + 5.0 % pure Al, N ₂
N-SC05B		99.5 % β-SiC + 0.5 % B, N ₂
N-SC50B		99.5 % β-SiC + 5.0 % B, N ₂



Figure 2. The relative density of SiC ceramics with 0.5 wt% and 5.0 wt% of different Al and B source sintered in Ar or N_2 atmosphere.

samples decreased with increasing the concentration of B in both sintering atmospheres. Whereas metal Al-added SiC achieved to be densifies reaching 94.3% of relative density in Ar atmosphere, their relative density was only 55.7% when SiC with 5.0 wt% pure Al was sintered in N₂ atmosphere. When the SiC was sintered using different Al source under Ar atmosphere, the relative density of A-SC05Al samples using nitrate source is 67.1%, which is showed better sintering performance than that using metal source with 54.2% relative density. Besides, the relative density of A-SC50Al samples using metal source is 94.3%, which is higher than using nitrate source with 86.5%. On the other hand, the relative density of A-SCB samples was similar with N-SCB samples, indicating the samples with B source was less affected by sintering atmosphere than those with Al source. However, comparing A-SCB with A-SCBC, A-SC05BC were able to densify SiC to 92.5% of relative density but that of A-SC05B was only 69.5%. Overall, the sintering atmosphere affected the density of Al-added SiC ceramics more than B-added ones, and the effect of additive form was obvious in both additions. The extremely low diffusivity of N in SiC (< 10⁻¹² cm²/s at 2550 °C) [10] is considered as the main reason to retard

the densification of SiC. Although the diffusivity of B is higher than Al in SiC above 1800 °C [10], the vapor transport of SiO and surface diffusion of Si [11] may be the rate controlling in the sintering process and thus resulted in a lower relative density for B-added samples.

3.2. Microstructure

The SEM images of pure Al-added and B-added SiC ceramics sintered in Ar atmosphere and N₂ atmosphere is shown in Figures 3 and 4, respectively. For the samples sintered in Ar atmosphere, the degree of grain growth increased with the additive content in both Al- and B-added SiC ceramics, and the grain growth was promoted into hexagonal platelet. The grain size of A-SC50Al-m was much larger than that of A-SC05Al-m, but the difference of grain size between A-SC05B and A-SC50B was rather small. On the other hand, SiC ceramics sintered in N₂ atmosphere had a more porous microstructure than the samples sintered in Ar atmosphere. Small degree of grain growth was promoted in N-SC05Al-m and N-SC50Al-m samples while N-SC05B and N-SC50B remained in microstructure with fine grains. Hexagonal platelets of SiC is commonly considered as the unique microstructure of α -SiC, and the presence of Al and B is found to be effective to promote grain growth and phase transformation of β to α -SiC, which agreed with the results of the samples sintered in Ar atmosphere. The mechanism of grain growth is thought to be originated from the presence of Si surface transport during sintering [11], which Si is possible to be produce from silica (SiO₂) in the starting SiC powder and the reaction of Al or B with SiC. The purity of β -SiC powder used in the present research was 93% with impurity of 0.1% crystalline Si and 1.7% oxygen. As for the sintering under N₂ atmosphere, due to the slow diffusion of N in SiC, it becomes the rate controlling process during sintering, resulted in the retard of densification [12]. N_2 is also considered to stabilize β -SiC (3C-SiC) [14], which agreed with the microstructure with fine grains observed in Figure 4. The mechanism of phase transformation is considered to



Figure 3. SEM images of (a) A-SC05Al-m, (b) A-SC50Al-m, (c) A-SC05B and (d) A-SC50B with higher magnification on the upper right.



Figure 4. SEM images of (a) N-SC05Al-m, (b) N-SC50Al-m, (c) N-SC05B and (d) N-SC50B with higher magnification on the upper right.

be result from the existence of stacking faults in SiC crystals, and the additive atoms are able to create stacking faults by diffusing through vacancies. Therefore, slower diffusivity gives less possibility for SiC to undergo phase transformation.

3.3. Composition of polytypes

The XRD profiles of pure Al- and B-added SiC ceramics sintered in Ar and N₂ atmosphere is illustrated in Figure 5. More each diffraction peaks attributed to α -SiC (6H-, 4Hand 15R-SiC) were identified in the samples sintered in Ar atmosphere than the samples sintered in N₂ atmosphere, and the intensity of those diffraction peaks became stronger in the samples sintered in Ar atmosphere. This result was in consistent with the observed microstructure, indicating the samples sintered in Ar atmosphere underwent phase transformation, and the samples sintered in N2 atmosphere remained in β-SiC (3C-SiC). Diffraction peaks for alumina (Al₂O₃), Si and Al were identified in A-SC50Al-m sample as secondary phases. Al₂O₃ is possible to be produced from the reaction of Al with SiO₂, and Si is the product from the reaction of Al with SiC [7]. On the other hand, aluminum nitride (AlN) was identified in N-SC50Al-m, and boron nitride (BN) was identified in both N-SC05B and N-SC50B samples. The presence of these nitrides in samples sintered under N_2 atmosphere could result from the reaction of Al or B additives with N_2 . The Gibbs free energy change of the reaction of Al or B with N_2 was calculated using MALT (first version, MALT group / Kagaku Gijutsu-Sha), and the values are both negative, indicating that these reactions are possible to happen.

The SiC polytype composition of each sample is shown in Figure 6. The polytype composition was analyzed by Rietveld method based on the XRD profiles of each sample. The agreement indices, S, was used to evaluate the accuracy of Rietveld refinement fitting on the raw XRD profile. The result of the analysis is considered reliable when the S value is smaller than 1.3 [13], and the analysis of every sample in this study met this requirement. Comparing the polytype composition of samples sintered in Ar and N₂ atmosphere, it is obvious that samples sintered in Ar atmosphere underwent high degree of phase transformation. The main phase of A-SC05Al-m, A-SC05B and A-SC50B was 6H-SiC, but the main phase of A-SC50Al-m was 4H-SiC (95.7%), indicating excess Al stabilized 4H-SiC while B stabilized 6H-SiC. The samples sintered in N₂ atmosphere showed higher content of 3C-SiC in B-added samples than Al-added samples. While Al and B stabilized α -SiC, and N stabilizes β -SiC, there is possible for reverse phase transformation to happen [14].



Figure 5. The XRD profiles of Al- or B-added SiC ceramics sintered in (a) Ar and (b) N_2 atmosphere.



Figure 6. The polytype composition analysed by Rietveld analysis of Al- or B-added SiC ceramics sintered in (a) Ar and (b) N₂ atmosphere.

The higher composition of 3C-SiC in samples sintered in N_2 atmosphere indicates the effect of N on β -SiC stabilization, and the lower composition of 3C-SiC in Al-added samples than B-added ones implied that Al affected the phase transformation of SiC than B.

3.4. Thermal conductivity

The thermal conductivity of Al- or B-added SiC ceramics sintered in Ar or N_2 atmosphere were measured from room temperature to 1000°C. The measured values were corrected for the porosity using the following equation based on the Maxwell-Eucken model [15];

$$\kappa_c = \kappa_m \left(\frac{1+V_d}{1-V_d}\right) \tag{3}$$

where κ_c and κ_m is the corrected and measured thermal conductivity, respectively, and V_d the volume fraction of pores. The κ_c represents the solid thermal conductivity without the existence of pores.

The κ_m of Al-and B-added SiC ceramics sintered in Ar and N_2 atmosphere is shown in Figure 7. Most of the sample sintered in Ar atmosphere showed higher κ_m than those sintered in N2 atmosphere. Among the samples sintered in Ar atmosphere, the κ_m of A-SC05B was 63 W/m·K at room temperature despite its total porosity was 30.5%. However, the κ_m of A-SC50Al-m was only 39 W/m·K at room temperature, despite having low porosity of 5.7%. After the porosity correction, the κ_c of A-SC05B still remained at a higher value (118 W/m·K) than A-SC50Alm, and the value of A-SC50B (87 W/ $m \cdot K$) became also higher than A-SC50Al-m. This indicates the effects of porosity and solid solution on the thermal conductivity, and the effect of solid solution on the thermal conductivity was seemed to stronger than porosity. The solubility of Al is higher than B in SiC lattice [8], which creates more defects in the lattice, and thus the degree of phonon scattering increased, resulting in lower κ_m . In this study, the analyzed occupancy of Al atoms in SiC lattice was higher than that of B atoms, which is able to explain the lower κ_m and κ_c of Al-added SiC ceramics. On the other hand, when different Al and B sources were used, the κ_m of A-SC50Al-n was 48 W/m·K and that of A-SC05BC was 99 W/m·K at room temperature. A-SC50Al-n possessed higher κ_m (48.2 W/m·K), than A-SC50Al-m (35.0 W/m·K) at room temperature despite its higher porosity of 13.5%. This indicates that porosity and solid solution may be less effective, and the higher content of secondary phases in A-SC50Al-m can be considered as the reason of the lower κ_m and κ_c .

When the sintering atmosphere changed from Ar to N_2 , the κ_m of most of the samples became lower, and this can be considered owing to the higher porosity of these samples. After the porosity correction, the κ_c of N-SC05Al-m became higher than A-SC05Al-m. Considering the occupancy of Al in SiC lattice of these two samples are in similar value, other factors such as secondary phases should be considered. From the analysis of XRD of N-SC05Al-m, no other secondary phases despite Al₂O₃ were identified. Nevertheless, there is possibility for Al to react with N₂ to produce AlN as mentioned above, and it could remain in amorphous or the small amount so that it was not identified by XRD. AlN generally shows high thermal conductivity, so its existence is believed to be the reason of the higher κ_c of N-SC05Al-m. On the other hand, the porosity of B-added SiC ceramics sintered in both atmospheres were almost similar but the k_m of those sintered in Ar atmosphere is three times higher. After porosity correction, the difference of κ_c of these samples remains the same. This can be considered due to the existence of BN, which was identified with the XRD profile. The thermal conductivity of BN is normally low, therefore, its presence as a secondary phase in SiC is possible to lower the overall κ_m and κ_c . However, further investigation is needed because the thermal conductivity of BN is anisotropic. For comparison, the κ_m of pure SiC sample sintered at the same temperature reported in our previous work [7] is 29.8 W/m·K. Overall, it is considered that both solid solution and secondary phases are important factors, but the latter would have higher impact due to the fact of its nano to micro scale whereas solid solution is in atomic scale.



Figure 7. The measured thermal conductivity (κ_m) of Al- or B-added SiC ceramics sintered in (a) Ar and (b) N₂ atmosphere.

3.5. Electrical resistivity

electrical resistivity determined at room The temperature for Al- and B-added SiC ceramics sintered in Ar or N₂ atmosphere is shown in Figure 8. For samples sintered in Ar atmosphere, Al-added SiC ceramics showed lower electrical resistivity (6.5 – 71.5 Ω ·cm) than B-added samples $(15.6 - 290.8 \,\Omega \cdot cm)$, and that of Al-added samples decreased with increasing the additive content but that of B-added samples increased with the additive content. For comparison, the electrical resistivity of pure SiC sample fabricated previously is 5.9 Ω cm, measured by four-point probe measurement. The low electrical resistivity of A-SC50Al-m can be considered to attribute to its low porosity of 5.7%. Although the porosity of A-SC05Al-m (45.8%) was slightly higher than A-SC05B (30.5%) and A-SC50B (44.7%), its electrical resistivity still remains at a low level. This is possible to be the effect of the difference in the solubility of Al and B atoms in SiC lattice. The result of analysis of occupancy of Al in SiC lattice is higher than B in this study, thus the acceptor level of Aladded samples might be higher than B-added ones, resulting in a lower electrical resistivity. When the Al source of Al-added to SiC ceramics sintered in Ar atmosphere was changed from nitrate to metal form, the electrical resistivity of A-SC50Al-n was 18.8 Ω ·cm, which was three times higher than that of A-SC50Al-m (6.5 Ω ·cm). The higher porosity of A-SC50Al-n (13.5%) can be considered as the reason of its higher electrical resistivity. As for changing the B source added to SiC ceramics sintered in Ar atmosphere, the electrical resistivity of A-SC05BC was 15.6 Ω cm, which was almost ten times lower than A-SC05B (162.2 Ω ·cm). The lower electrical resistivity of A-SC05BC can be though as owing to its lower porosity of 7.5%, however, its porosity was close to that of A-SC50Al-m (5.7%), but its electrical resistivity was still 2.5 times higher than A-SC50Al-m, once again showing the effect of solid solution formed by Al or B atoms with SiC lattice.

On the other hand, the electrical resistivity of all Aland B-added SiC ceramics sintered in N₂ atmosphere increased with the additive content. The electrical resistivity of N-SC50Al-m (335.6 Ω ·cm) was higher than the samples sintered in Ar atmosphere, this can be the effect of higher porosity. However, its porosity, 44.3%, was about 3% lower than N-SC05Al-m and about 1% lower than A-SC05Al-m, but its electrical resistivity was about 10 times higher than N-SC05Al-m and nearly 17 times higher than A-SC05Al-m. Therefore, porosity is not the only factor affecting electrical resistivity. The presence of AlN, which is an insulator, identified with the XRD profile may be the reason of such high electrical resistivity of N-SC50Al-m. In addition, Al and N are p-type and ntype dopants in SiC lattice, respectively; therefore, the acceptors formed by Al doping is possible to compensate with carriers created by N doping, and thus increasing the electrical resistivity. Interestingly, B-added SiC ceramics possessed similar porosity in both sintering atmospheres, but their electrical resistivity behaved differently, and the samples sintered in N2 atmosphere showed lower electrical resistivity. The main SiC polytype of N-SC05B and N-SC50B was 3C-SiC, and that of A-SC05B and A-SC50B was 6H-SiC; therefore, the electrical conduction mechanism may be different in different crystal structure of SiC. Besides, the compensation of acceptors and carries is also possible to happen in B-added SiC ceramics but owing to the higher solubility of N in SiC lattice than B [8], the electronic conduction may shift to n-type and create a lower electrical resistivity.

4. Conclusion

The effect of sintering atmosphere and additive form on the thermal and electrical properties of SiC ceramics are conclude as follows:

(1) Effect of sintering atmosphere: The sintering behavior of Al-added SiC ceramics was strongly affected by the different sintering atmosphere of Ar and N₂. The grain growth and phase transformation was both retarded by the N₂ atmosphere in both Al- and B-added SiC ceramics. The effect of sintering atmosphere is found out to be more significant on electrical properties of the SiC ceramics sintered in this study than thermal properties. Solid solutions and secondary phase of nitrides are thought to both affect the thermal conductivity and electrical resistivity in this study.



(2) Effect of additive forms: The difference of sintering

Figure 8. The electrical resistivity at room temperature of Al- or B-added SiC ceramics sintered in (a) Ar and (b) N₂ atmosphere.

behavior of B-added SiC ceramics was more obvious than that of Al-added samples when changing the additive form from B to B_4C and pure Al to Al nitrate. The resulted difference in density further affected the thermal conductivity and electrical resistivity of these SiC ceramics.

Solid solution is found to be able to control both thermal conductivity and electrical resistivity. By using additive that has high solubility in SiC is able to produce low thermal conductivity and relatively low electrical resistivity, and is expected to produce desired ZT value for high temperature thermoelectric performance.

Acknowledgements

This work was supported by Japan Science and Technology Agency (JST) SPRING, Grant Number JPMJSP2106.

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