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RESEARCH ARTICLE

POROUS SiO_2-SiC BASED CERAMICS WITH LOW THERMAL CONDUCTIVITY

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Abstract. Porous SiO_2–SiC-based ceramics were developed to achieve superior thermal insulation and mechanical performance for high-temperature applications such as thermal protection and energy conversion systems. In this study, nano-sized SiO_2, nano-sized SiC, and carbon black powders were used to fabricate porous SiO_2–SiC ceramics by sintering in air at 700–1000 °C. The effects of nano-SiC content (0–35 wt%) and sintering temperature on porosity, thermal conductivity, and compressive strength were systematically investigated. Increasing the nano-SiC content and sintering temperature led to enhanced partial densification of the struts due to silica bonding, resulting in decreased porosity from 77.1% to 69.5%. The lowest thermal conductivity of 0.043 W/m·K was achieved for samples containing 10 wt% nano-SiC sintered at 700 °C, attributed to the high interfacial thermal resistance at SiO_2–SiC interfaces. The compressive strength of porous SiO_2-SiC based ceramics increased by 5.4 – 6.9 times with an increase in sintering temperature and the nano-SiC content from 0 to 35 wt% and remained significantly higher than that of previously reported porous SiC ceramics. The improved thermal insulation and mechanical strength were attributed to strong silica promoted interparticle bonding and the formation of SiO_2 core/SiC shell structures. These findings demonstrate that the newly developed porous SiO_2–SiC ceramics possess a promising combination of low thermal conductivity and high strength for advanced high-temperature applications.

Keywords: porous ceramic, SiO_2-SiC, porosity, thermal conductivity, compressive strength.

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ҒЫЛЫМИ МАҚАЛА

ТӨМЕН ЖЫЛУ ӨТКІЗГІШТІГІ БАР SiO_2-SiC НЕГІЗІНДЕГІ КЕУЕКТІ КЕРАМИКА

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Аңдатпа. Кеуекті SiO_2–SiC негізіндегі керамикалар жоғары температуралы қолданбаларда, мысалы, жылулық қорғаныс және энергия түрлендіру жүйелерінде жоғары жылу оқшаулау және механикалық қасиеттерге қол жеткізу үшін жасалды. Бұл зерттеуде ауада 700–1000 °C температурада күйдіру арқылы кеуекті SiO_2–SiC керамикасын алу үшін наноөлшемді SiO_2, наноөлшемді SiC және көміртек қара ұнтағы пайдаланылды. Нано-SiC мөлшерінің (0–35 масс.%) және күйдіру температурасының кеуектілікке, жылуөткізгіштікке және қысу беріктігіне әсері жүйелі түрде зерттелді. Нано-SiC мөлшерін және күйдіру температурасын арттыру кремнезем байланысының әсерінен қаңқаның жартылай тығыздалуын күшейтіп, кеуектіліктің 77.1%-дан 69.5%-ға дейін төмендеуіне әкелді. Ең төмен жылуөткізгіштік 0.043 Вт/м·К мәні 10 масс.% нано-SiC бар және 700 °C-та күйдірілген үлгі үшін анықталды, бұл SiO_2–SiC интерфейстеріндегі жоғары шекаралық жылу кедергісімен түсіндірілді. Кеуекті SiO_2–SiC негізіндегі керамикалардың қысу беріктігі күйдіру температурасы мен нано-SiC мөлшерін 0-ден 35 масс.% дейін арттырғанда 5.4–6.9 есеге өсті және бұрын хабарланған кеуекті SiC керамикаларына қарағанда әлдеқайда жоғары болды. Жақсартылған жылу оқшаулау және механикалық беріктік кремнеземнің бөлшектер арасындағы күшті байланысын және SiO_2 өзек/SiC қабық құрылымдарының түзілуін қамтамасыз етті. Бұл нәтижелер жаңадан әзірленген кеуекті SiO_2–SiC керамикаларының төмен жылуөткізгіштік пен жоғары беріктік комбинациясын иеленіп, жоғары температуралы заманауи қолданбалар үшін үлкен әлеуетке ие екенін көрсетеді.

Түйін сөздер: кеуекті керамика, SiO_2–SiC, кеуектілік, жылуөткізгіштік, қысу беріктігі.

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НАУЧНАЯ СТАТЬЯ

ПОРИСТАЯ КЕРАМИКА НА ОСНОВЕ SiO_2-SiC С НИЗКОЙ ТЕПЛОПРОВОДНОСТЬЮ

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Аннотация. Пористые керамики на основе SiO_2–SiC были разработаны для достижения превосходных теплоизоляционных и механических свойств при высокотемпературных применениях, таких как системы тепловой защиты и преобразования энергии. В данном исследовании для изготовления пористых керамик SiO_2–SiC использовались наноразмерные порошки SiO_2, SiC и сажи, которые спекались на воздухе при температуре 700–1000 °C. Влияние содержания нано-SiC (0–35 мас. %) и температуры спекания на пористость, теплопроводность и прочность при сжатии было изучено систематически. Повышение содержания нано-SiC и температуры спекания усиливало частичное уплотнение каркаса за счёт связывания кремнеземом, что приводило к уменьшению пористости с 77,1 % до 69,5 %. Наименьшая теплопроводность 0,043 Вт/м·К была достигнута для образцов, содержащих 10 мас. % нано-SiC, спечённых при 700 °C, что объясняется высокой межфазной тепловой сопротивляемостью на границах SiO_2–SiC. Прочность при сжатии пористых керамик на основе SiO_2–SiC увеличивалась в 5,4–6,9 раза с повышением температуры спекания и содержания нано-SiC от 0 до 35 мас. % и оставалась значительно выше, чем у ранее сообщённых пористых керамик SiC. Улучшенные теплоизоляционные и механические свойства объясняются прочным межчастичным связыванием, обусловленным кремнеземом, и формированием структур типа SiO_2-ядро/SiC-оболочка. Полученные результаты показывают, что новые пористые керамики SiO_2–SiC обладают перспективным сочетанием низкой теплопроводности и высокой прочности для передовых высокотемпературных применений.

Ключевые слова: пористая керамика, SiO_2–SiC, пористость, теплопроводность, прочность при сжатии.

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CONFLICT OF INTEREST

The authors state that there is no conflict of interest.

АЛҒЫС / ҚАРЖЫЛАНДЫРУ КӨЗІ

Зерттеу Қазақстан Республикасы Ғылым және жоғары білім министрлігі Ғылым комитетінің ЖТН AP19174518 гранттық қаржыландыру шеңберінде жүргізілді.

МҮДДЕЛЕР ҚАҚТЫҒЫСЫ

Авторлар мүдделер қақтығысы жоқ деп мәлімдейді.

БЛАГОДАРНОСТИ/ИСТОЧНИК ФИНАНСИРОВАНИЯ

Исследование проводилось в рамках грантового финансирования Комитета науки Министерства науки и высшего образования Республики Казахстан ИРН AP19174518.

КОНФЛИКТ ИНТЕРЕСОВ

Авторы заявляют, что конфликта интересов нет.

1 INTRODUCTION

Advanced materials used in thermal protection systems, energy conversion systems, and high-temperature industrial installations must have properties such as extremely low thermal conductivity and structural stability at high temperatures. In thermal protection systems (e.g., in aviation, spacecraft, furnaces, and turbines), the primary role of the material is to suppress heat transfer from the high-temperature environment (flame, plasma, or hot gases) to the protected structures or equipment. The use of a material with high thermal conductivity in thermal protection systems is unacceptable, as heat can easily pass through it, which may lead to overheating of the internal parts of the structure and, consequently, to its failure. Minimizing heat losses is critical in energy conversion systems (such as gas turbines and thermoelectric generators). Materials with intrinsically low thermal conductivity maintain a stable thermal gradient (temperature difference), thereby enabling more efficient energy conversion. In high-temperature industrial facilities, heating conditions are often non-uniform. Materials with low thermal conductivity help localize heat within the desired zone, mitigating thermal stress and reducing the risk of structural failure. In aerospace applications, even a temperature rise of several hundred degrees can have catastrophic consequences. Therefore, materials must not only withstand extreme temperatures but also provide effective thermal insulation for internal components.

Porous SiC-SiO_2 ceramics possess unique properties such as chemical stability, excellent oxidation resistance, and mechanical integrity (**Sheng et al., 2019; Chen & Miyamoto, 2014**) [25,31]. Owing to their properties porous SiC-SiO_2 ceramics used for various applications such as catalyst supports (**She et al.,2003; Chun & Kim, 2005**) [28,27], membranes (**Sheng et al., 2019**) [25], hot-gas filters (Dey et al., 2011) [26], electromagnetic radiation absorbers (**Yuan, Cheng and Zhang, 2016**) [29], and microwave absorbers (**Yuan et al., 2016**) [30]. The incorporation of SiC into SiO_2 or SiO_2 into SiC creates more interfaces between the two phases, resulting in enhanced phonon scattering. Thus, porous SiO_2-SiC based ceramics have high potential for thermal insulation applications.

2 LITERATURE REVIEW

Thermal conductivities of porous SiC-based ceramics have been studied widely by many researchers which elaborated several methods to lower thermal conductivity of porous SiC-based ceramics: (1) porosity maximization by gel-freezing **(Yoon et al., 2007; Fukushima et al., 2010; Fukushima et al., 2014)**, direct foaming (**Kim et al., 2005; Jana et al., 2017**) [11,12], filament printing and partial sintering (**Gomez-Gomez et al., 2019**), sol gel process followed by supercritical drying (**Kong et al., 2014; Su et al., 2018**), compression molding (**Manoj Kumar et al., 2011**), chemical vapor infiltration and reaction (**Streitwieser et al., 2005**); (2) incorporating an in-situ synthesized thermally insulating secondary phase into porous SiC ceramics by partial oxidation of nano-sized SiC particles (**Malik et al., 2020**); (3) fabrication of nano-structures with nano-porosity or hierarchical porosity (**Wan et al., 2018**). Recently, **Jana et al., 2017** investigated the effects of direct foaming of SiC slurries followed by gel casting and sintering on the thermal and mechanical properties of SiC foams and reported that the thermal conductivity and compressive strength were 4.3 W/mK and 1.7 MPa, respectively, at a porosity of 89%. Extremely low thermal conductivity of 0.026 W/mK showed SiC nanowire aerogels which have been processed by sol-gel process and subsequent supercritical drying at a porosity of 99,8 % (**Su et al., 2018**). SiC foam fabricated by gel-freezing method exhibited the thermal conductivity of 0.054 W/mK at 98% porosity (**Fukushima & Yoshizawa, 2014**). Introduction of polysiloxane derived silica into nano-SiC ceramics decreased the thermal conductivity to 0.047 W/mK and showed minimal loss of compressive strength of 1.8 MPa at a porosity of 72.4% (**Malik et al., 2020**). **Kim et al., 2020** studied the thermal and mechanical properties of silica-bonded porous nano-SiC ceramics and reported that the thermal conductivity and compressive strength were 0.057 and 2.5 MPa, respectively, at 70.2% porosity.

In this study several strategies were established to obtain very low thermal conductivity and maximized mechanical strength in porous SiO_2-SiC ceramics, compared to the previous studies: (1) addition of nano-SiC into nano-SiO_2 to create SiO_2-SiC interfaces, (2) addition of nano-sized carbon template into nano-SiO_2 powder to create pores, (3) sintering at the different temperatures from 700°C to 1000°C.

The effect of nano-SiC content and sintering temperatures on the porosity, microstructure, thermal conductivity, and compressive strength of the newly developed porous SiO_2-SiC ceramics were investigated.

3 METHODS AND MATERIALS

Porous SiO_2-SiC based ceramics were prepared using commercially available nano-sized SiO_2 (~25 nm, Aerosil 300, Degussa AG, Hanau-Wolfgang, Germany), nano-sized β-SiC (~50 nm, 97.5%, N&A Materials, Inc., USA), and nano-sized carbon black (~75 nm, N774, OCI Company, Ltd., Korea) as a sintering materials. Nano-sized SiO_2 powder (Slurry 1), β-SiC powder (Slurry 2), and carbon black (Slurry 3) were each dispersed in distilled (DI) water using SiC balls. Slurry 1 was prepared by conventional ball milling for 24 h, while Slurries 2 and 3 were processed by planetary ball milling for 2 h. Subsequently, all three slurries were combined and mixed using conventional ball milling for an additional 2 h. The resulting mixture was dried and then milled again with organic binders in ethanol for 2 hours, using SiC balls and a polypropylene jar. Four different batches were prepared by varying the β-SiC content from 0 to 35 wt%: S0, S10, S20, and S35, where the number indicates the wt% of β-SiC. The carbon content was kept constant at 40 wt%. Afterwards, the mixtures were dried overnight in an oven at 70 °C. The dried powders were then ground and granulated by passing through a 120-mesh sieve. The resulting powders were uniaxially pressed under 15 MPa into green compacts of two sizes: $7 \times 7 \times 14$ mm^3 for compressive strength tests, and $10 \times 10 \times 2.5$ mm^3 for thermal conductivity measurements. green compacts were sintered in air at 700–1000 °C for 2 h.

The sintered specimens were designated as follows: for example, S0-7, S10-7, S20-7, and S35-7, where the number after the dash (7) indicates the sintering temperature of 700 °C. A similar notation was used for samples sintered at 800 °C, 900 °C, and 1000 °C.

The theoretical density of the porous SiO_2-SiC ceramics was calculated using the rule of mixtures according to the following formula:

$$\rho_{th} = \rho_{SiO_2} V_{SiO_2} + \rho_{SiC} V_{SiC} \qquad (1)$$

where, ρ_{SiO_2} and ρ_{SiC} are the theoretical densities of silica 2.196 (gcm^{-3}) and SiC (3.216 gcm^{-3}), respectively. V_{SiO_2} and V_{SiC} denotes the volume fraction of silica and SiC which calculated from change of the weight after sintering in air, respectively. The SiO_2 content calculated from the weight change after sintering by the following reaction:

$$SiC + O_2 \rightarrow SiO_2 + CO_2 \qquad (2)$$

$$C + O_2 \rightarrow CO_2 \qquad (3)$$

The bulk density of the sintered samples was calculated from the weight to volume ratio. The porosity of the samples obtained by following equation:

$$P = 1 - \frac{\rho_b}{\rho_{th}} \times 100 \qquad (4)$$

where, P and ρ_b are the porosity (%) and bulk density of the porous SiO_2-SiC ceramics, respectively.

Microstructural analysis was carried out using scanning electron microscopy (SEM, S4300, Hitachi Ltd., Hitachi, Japan). Compressive strength was evaluated using an Instron 3344 testing machine (Instron Inc., Norwood, MA, USA) at a constant crosshead speed of 0.5 mm/min. The compressive strength of porous SiO_2–SiC ceramics was tested six times for each sample type to ensure reproducibility. Thermal diffusivity and heat capacity were determined using the laser flash method (LFA 467; NETSCH GmbH, Selb, Germany), with a thin graphite coating applied to the sample surfaces prior to measurement. Each sample was tested three times in an argon atmosphere. Thermal conductivity was calculated using the following equation [100]:

$$\kappa = \alpha \rho C_p \qquad (5)$$

where ρ, α, and C_p denotes the sintered density, thermal diffusivity, and heat capacity, respectively.

Table 1

Batch composition and sintering condition of SiO_2-SiC ceramics

Sample designation	Batch composition (wt%)			Sintering condition	Remark
	SiO_2	β-SiC	CB		
S0	60	-	40	700°C/2h/Air 800°C/2h/Air 900°C/2h/Air 1000°C/2h/Air	0wt% SiC
S10	54	6	40		10wt% SiC
S20	48	12	40		20wt% SiC
S35	39	21	40		35wt% SiC

4 RESULTS AND DISCUSSION

4.1 Porosity and microstructure

Figure 1 shows the porosity of porous SiO_2–SiC-based ceramics as a function of the initial nano-SiC content, sintered at 700 °C–1000 °C in air. As the nano-SiC content increased from 0 wt% to 35 wt%, the porosity gradually decreased from 77.1% to 75.0% for samples sintered at 700 °C, from 76.9% to 74.3% at 800 °C, and from 75.4% to 72.2% at 900 °C. However, at 1000 °C, the porosity initially increased from 67.3% to 70.7% as the nano-SiC content increased from 0 wt% to 10 wt%, and then gradually decreased to 70.6% and 69.5% with further increases to 20 wt% and 35 wt%, respectively. The addition of nano-SiC further increased the density due to partial densification promoted by silica. The higher silica content enhances interparticle bonding, pulling adjacent particles closer together and resulting in partial densification.

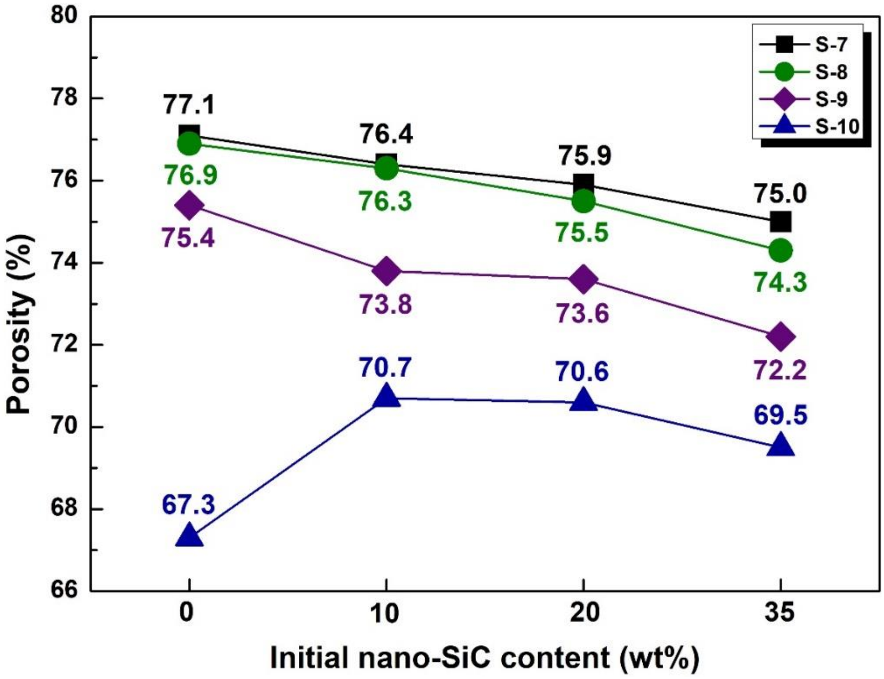

Figure 1 – Porosity of porous SiO_2-SiC based ceramics

The S0 samples exhibited a decrease in porosity from 77.1% to 67.3% when the sintering temperature increased from 700 °C to 1000 °C, respectively. This is attributed to increased partial densification due to increased silica content at high temperatures. The increased silica content causes strong inter-particle bonding, thus pulling the adjacent particles closer and causes partial densification. The increased silica content gives rise to strong inter-particle bonding, pulling adjacent particles closer to each other, and leads to the enhanced densification of the porous ceramics by viscous flow. The S10, S20, and S35 samples also exhibited a decrease in porosity from 76.4% to 70.7%, from 75.9% to 70.6%, and from 75.0% to 69.5% when the sintering temperature increased from 700 °C to 1000 °C, respectively.

Figure 2 shows typical microstructure of porous SiO_2-SiC ceramics sintered at 700 °C for 2 h in air. The SEM micrographs confirm a decrease in porosity with increasing nano-SiC content from 0 wt% to 35 wt% at a given sintering temperature. The microstructure exhibits small clusters of particle aggregates bonded together and two types of pores: inter-aggregate pores and intra-aggregate pores.

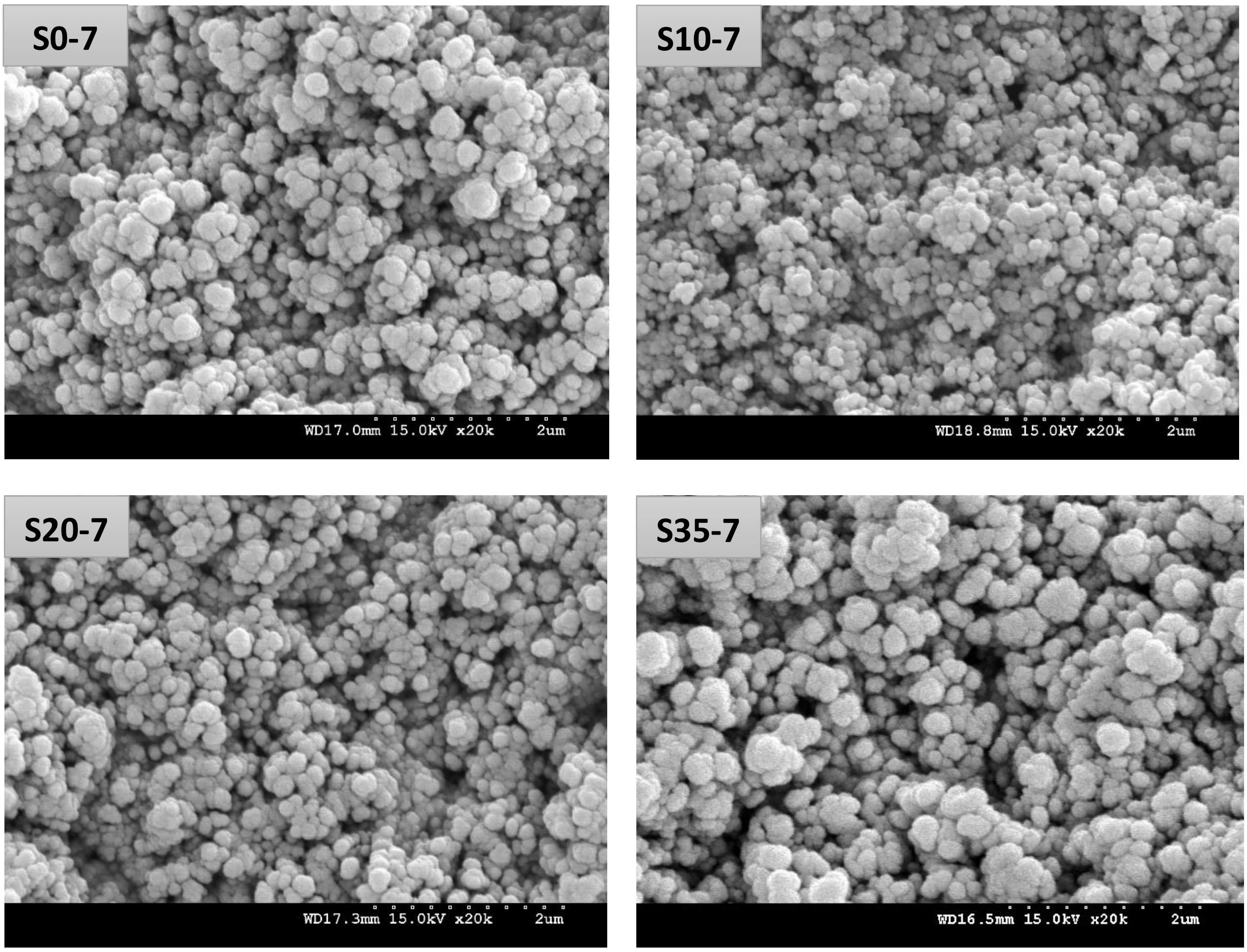

Figure 2 – The typical microstructure of porous SiO_2-SiC ceramics sintered at 700 °C for 2 h in air.

4.2 Thermal conductivity

Figure 3 exhibited the thermal conductivities of porous SiO_2-SiC based ceramics. The lowest thermal conductivity was achieved for sample S0-7 (porous SiO_2-SiC based ceramics with 0 wt% of nano-SiC content sintered at 700 °C). The thermal conductivity increased from 0.036 to 0.050 W/mK, 0.045 to 0.056 W/mK, and 0.050 to 0.059 W/mK with increasing the initial nano-SiC powder content from 0 to 35 wt% for S-7, S-8, and S-9 samples, respectively. However, the thermal conductivity of S-10 sample decreased from 0.127 to 0.076 W/mK when initial nano-SiC content increased from 0 to 20 wt%, respectively. Thermal conductivity of S10 (porous SiO_2-SiC based ceramic with 10 wt% of nano-SiC) sample increased from 0.043 to 0.087 W/mK with an increase of sintering temperature from 700 °C to 1000 °C, respectively. Generally, thermal conductivity increases with decreasing porosity, and the porous SiO_2-SiC based ceramics sintered at 700 °C, 800 °C, and 900 °C followed this trend.

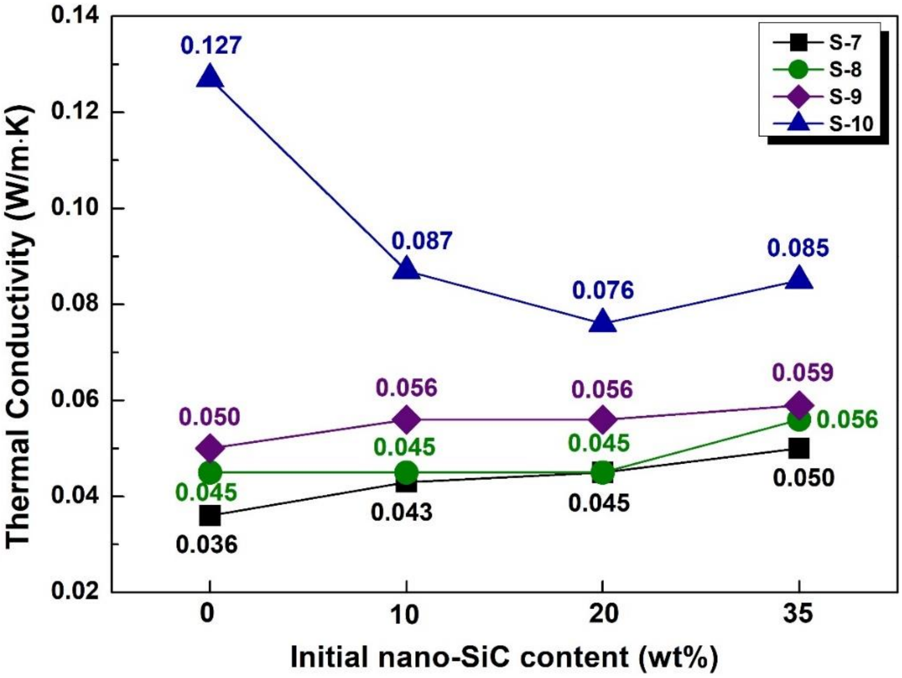

Figure 3 – Thermal conductivity of porous SiO_2-SiC based ceramics

These results suggest that the addition of nano-SiC into nano-SiO_2 increases thermal conductivity. The increase of thermal conductivity was attributed to the decrease of porosity. The porosity decreased with increasing the initial nano-SiC content was due to the partial densification of the porous SiO_2-SiC based ceramics by viscous flow and/or higher packing density of the green body.

Thermal conductivities of newly developed porous SiO_2-SiC ceramics were 0.045 W/mK (S10-8) and 0.043 W/mK (S10-7) which are one order of magnitude lower than that of porous nano-SiC ceramics 0.14 W/mK at the similar porosity of ~76.3% (**Wan & Wang, 2018**). The thermal conductivity of porous SiO_2-SiC ceramic with 20 wt% of nano-SiC sintered at 900 °C (S20-9) was lower (0.056 W/mK) than that the reported literature data (0.068 W/mK) at the similar porosity of 73.5% (**Wan, Gao and Wang, 2017**).

These results suggest that the addition of nano-SiC content into nano-SiO_2 could decrease the thermal conductivity of the porous SiO_2-SiC ceramic which was attributed to the increase of interfacial thermal resistance at the SiO_2-SiC interfaces.

4.3 Compressive strength

Compressive strength of porous SiO_2-SiC based ceramics is shown in **Figure 4**. The compressive strength of porous SiO_2-SiC based ceramics increased with increasing sintering temperature. For example, the thermal conductivity of S0, S10, S20, and S35 increased from 3.0 to 20.7 MPa, from 2.9 to 15.7 MPa, from 2.5 to 10.9 MPa, and from1.9 to 6.9 MPa with increasing temperature from 700 °C to 1000 °C, respectively. The compressive strength of porous SiO_2-SiC based ceramics decreased from 20.7 to 6.9 MPa, 9.3 to 3.4 MPa, 4.3 to 2.9 MPa, and 3.0 to 1.9 MPa with increasing initial nano-SiC content from 0 wt% to 35 wt% for S-10, S-9, S-8, and S-7 samples, respectively.

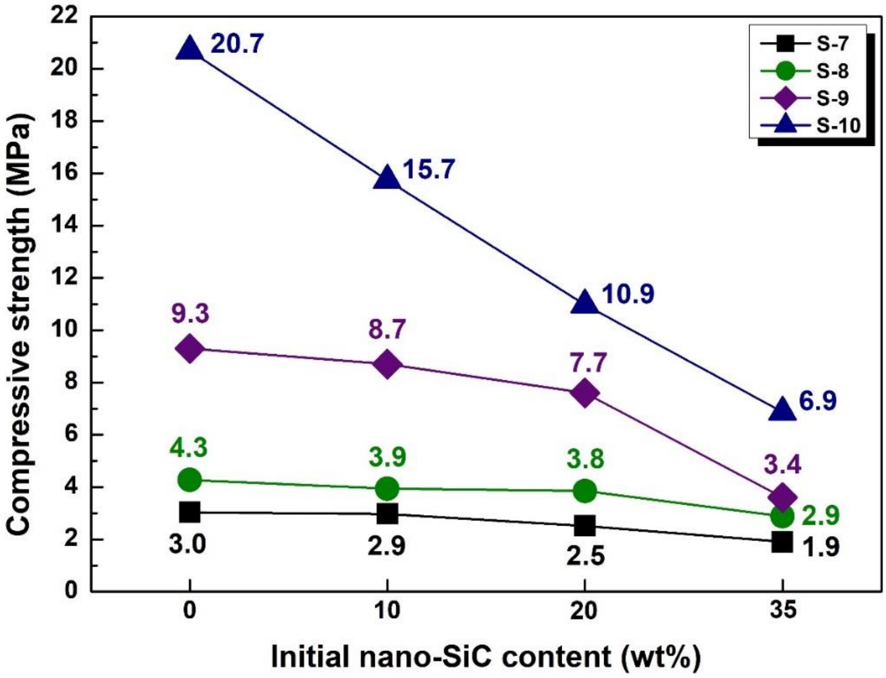

Figure 4 – Compressive strength of porous SiO_2-SiC based ceramics as a function of initial nano-SiC content

The compressive strengths of porous SiO_2-SiC based ceramics were 2 and 2,4 times higher (2.9 MPa for S10-7 and 3.9 MPa for S10-6, respectively) than that of porous nano-SiC ceramics (1.6 MPa) at a same porosity of 76.3% (**Wan & Wang, 2018**). The S20-10 sample exhibited the compressive strength of 10.9 MPa which was 4 times higher than that of porous nano-SiC ceramic reported in the literature data at the porosity of ~70.4% (**Kim et al., 2020**). Additionally, S35-9 sample (porous SiO_2-SiC based ceramic sintered with 35 wt% of nano-SiC at 900°C) showed the compressive strength of 3.4 MPa which is approximately 2 times higher than the literature data (1.8 MPa) at porosity of ~72.3% (**Malik et al., 2020**).

The present results suggest that the porous SiO_2-SiC based ceramics (S) with very low thermal conductivities of 0,043 – 0,056 W/mK exhibited much higher compressive strengths of 2,9 – 3,9 MPa than the previously reported SiC based porous ceramics. The highest strength and lower thermal conductivity of newly developed porous SiO_2-SiC based ceramics was attributed to the high strength interparticle bonding ensured by silica bonding and high interfacial thermal resistance obtained by introducing SiO_2 core / SiC shell structure.

5 CONCLUSIONS

The effects of nano-SiC content and sintering temperature on the thermal conductivity and compressive strength of porous SiO_2-SiC ceramics were investigated. The important findings are summarized as follows:

(1) The decreased porosity of the porous SiO_2–SiC ceramics with increasing nano-SiC content and sintering temperature was attributed to the enhanced partial densification of the struts caused by the increased silica content.

(2) The addition of nano-SiC into a nano-SiO_2 and nano-sized carbon template mixture is beneficial for increasing the thermal resistance due to the generation of additional interfaces, resulting in an extremely low thermal conductivity in porous SiO_2-SiC based ceramics reaching as low as 0.043 W/mK at 76.4 % porosity.

(3) The compressive strength of porous SiO_2-SiC based ceramics increased by 5.4 – 6.9 times with an increase in the nano-SiC content from 0 to 35 wt% when sintered at 700–1000 °C.

(4) The typical thermal conductivity and compressive strength values of thr porous SiO_2-SiC ceramics at 0.043 W/mK and 2.9 MPa, respectively.

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