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RESEARCH ARTICLE

EFFECT OF ANNEALING TEMPERATURE AND ATMOSPHERE ON THE PROPERTIES POROUS SIO_2–SIC CERAMICS

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Abstract. *This study investigates the influence of annealing at 1000 °C for 1 h in argon and air atmospheres on the microstructure and properties of porous SiO_2–SiC ceramics containing 0–35 wt% nano-SiC. The porosity increased slightly with the addition of nano-SiC, from 75.2 % to 76.0 % for samples annealed in argon, and from 64.8 % to 67.5 % for those treated in air. The higher porosity in the air-annealed specimens is attributed to partial oxidation of SiC, which produces gaseous by-products that promote pore formation. Thermal conductivity decreased from 0.152 to 0.122 $W\ m^{-1}\ K^{-1}$ in the argon series and from 0.058 to 0.045 $W\ m^{-1}\ K^{-1}$ in the air series, owing to enhanced phonon scattering by pores and oxidation-derived SiO_2 layers. Compressive strength showed the opposite trend, declining from 24.8 to 14.1 MPa in argon and from 10.6 to 4.0 MPa in air as SiC content increased. These results demonstrate that annealing atmosphere strongly affects pore evolution, oxidation behavior, and interfacial bonding, thus controlling the balance between thermal insulation and mechanical stability in porous SiO_2–SiC ceramics.*

Keywords: *porous ceramics; SiO_2–SiC composites; annealing atmosphere; thermal conductivity; compressive strength*

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ҒЫЛЫМИ МАҚАЛА

КЕУЕКТІ SIO_2–SIC КЕРАМИКАСЫНЫҢ ҚАСИЕТТЕРІНЕ КҮЙДІРУ ТЕМПЕРАТУРАСЫ МЕН АТМОСФЕРАСЫНЫҢ ӘСЕРІ

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Аңдатпа. *Бұл зерттеу 1000 °C температурада 1 сағат бойы аргон және ауа атмосфераларында өткізілген күйдірудің 0–35 мас.% нано-SiC қамтылған кеуекті* SiO_2*–SiC керамикасының микроқұрылымы мен қасиеттеріне әсерін қарастырады. Нано-SiC мөлшері артқан сайын кеуектілік сәл өсті: аргонда күйдірілген үлгілерде — 75,2 %-дан 76,0 %-ға дейін, ал ауада өңделген үлгілерде — 64,8 %-дан 67,5 %-ға дейін. Ауада күйдірілген үлгілердің кеуектілігінің жоғары болуы SiC-тің жартылай тотығуымен және кеуек түзілуін күшейтетін газ тәрізді өнімдердің бөлінуімен түсіндіріледі. Жылуөткізгіштік аргон сериясында 0,152-ден 0,122 Вт/м·К-ге дейін және ауа сериясында 0,058-ден 0,045 Вт/м·К-ге дейін төмендеді, бұл фонондардың кеуек беттерінде шашырауы мен* SiO_2 *тотығу қабаттарының түзілуімен байланысты. Қысу беріктігі керісінше төмендеді: аргонда 24,8-ден 14,1 МПа-ға, ауада 10,6-дан 4,0 МПа-ға дейін. Нәтижелер күйдіру атмосферасының кеуек эволюциясына, тотығу процестеріне және фазааралық байланыстарға айтарлықтай әсер ететінін, осылайша кеуекті* SiO_2*–SiC керамикасының жылуоқшаулау мен механикалық тұрақтылық арасындағы тепе-теңдікті басқаратынын көрсетеді.*

Түйін сөздер: *кеуекті керамика;* SiO_2*–SiC композиттері; күйдіру атмосферасы; жылуөткізгіштік; сығылу беріктігі.*

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НАУЧНАЯ СТАТЬЯ

ВЛИЯНИЕ ТЕМПЕРАТУРЫ И АТМОСФЕРЫ ОТЖИГА НА СВОЙСТВА ПОРИСТОЙ КЕРАМИКИ SIO_2–SIC

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Аннотация. *В данной работе исследовано влияние отжига при 1000 °C в течение 1 ч в атмосфере аргона и воздуха на микроструктуру и свойства пористой керамики на основе SiO_2–SiC, содержащей 0–35 мас.% нано-SiC. С увеличением содержания нано-SiC пористость несколько возрастала: с 75,2 % до 76,0 % для образцов, отожжённых в аргоне, и с 64,8 % до 67,5 % для образцов, обработанных в воздухе. Более высокая пористость при отжиге в воздухе связана с частичным окислением SiC, сопровождающимся выделением газообразных продуктов, способствующих образованию пор. Теплопроводность уменьшалась с 0,152 до 0,122 Вт/м·К в серии, отожжённой в аргоне, и с 0,058 до 0,045 Вт/м·К в серии, обработанной в воздухе, что объясняется усиленным рассеянием фононов на порах и образованием оксидных слоёв SiO_2. Прочность при сжатии, напротив, снижалась с 24,8 до 14,1 МПа в аргоне и с 10,6 до 4,0 МПа в воздухе при увеличении содержания SiC. Полученные результаты показывают, что атмосфера отжига существенно влияет на эволюцию пор, процессы окисления и межфазное взаимодействие, определяя соотношение между теплоизоляционными и механическими свойствами пористой керамики SiO_2–SiC.*

Ключевые слова: *пористая керамика; композиты SiO_2–SiC; атмосфера отжига; теплопроводность; прочность при сжатии*

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CONFLICT OF INTEREST

The authors state that there is no conflict of interest.

The authors declare that no generative artificial intelligence technologies or AI-based tools were used in the preparation of this article.

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.

Авторлар мақаланы дайындау барысында генеративті жасанды интеллект технологиялары мен жасанды интеллектке негізделген технологияларды пайдаланбағанын 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.

Авторы заявляют о том, что при подготовке статьи не использовались технологии генеративного искусственного интеллекта и технологии, основанные на искусственном интеллекте.

1 INTRODUCTION

Porous ceramics have become an essential class of advanced materials due to their unique combination of low density, high thermal stability, and chemical inertness, which make them suitable for use in extreme environments. These materials are widely employed in high-temperature thermal insulation, catalyst supports, filtration membranes, refractory linings, and energy-efficient construction systems (**She et al., 2003**). Their microstructure, consisting of interconnected pores within a rigid ceramic matrix, provides a balance between mechanical integrity and desired functional properties such as low thermal conductivity and high permeability (**Dey et al., 2011**). The key advantage of porous ceramics lies in their ability to maintain dimensional and chemical stability at temperatures exceeding 1000 °C while exhibiting adjustable porosity levels and lightweight characteristics.

Among various porous ceramic systems, silicon carbide (SiC) and silicon dioxide (SiO_2) based composites stand out for their synergistic combination of thermal and mechanical properties. SiC possesses outstanding characteristics such as high hardness, strong covalent bonding, superior thermal conductivity (~120 $W\ m^{-1}\ K^{-1}$ for dense SiC), and excellent oxidation resistance at elevated temperatures (**Yuan et al., 2016**). Conversely, SiO_2 offers low intrinsic thermal conductivity (~1.4 $W\ m^{-1}\ K^{-1}$), a relatively low sintering temperature, and good processability, making it a valuable component for forming porous structures (**Yuan et al., 2016a**). When combined, the SiO_2–SiC composite system integrates the thermal insulation capability of silica with the thermal stability and mechanical robustness of SiC, yielding a promising material for next-generation thermal barrier and insulating applications (**Kim et al., 2005**).

In porous SiO_2–SiC ceramics, microstructural evolution during fabrication and subsequent heat treatment plays a decisive role in determining final performance. The microstructure is governed by factors such as sintering temperature, time, particle size, and most critically, the annealing atmosphere (**Jana et al., 2017**). During thermal treatment, SiC particles can undergo partial oxidation. This reaction leads to the formation of a thin SiO_2 layer on SiC surfaces and the generation of gaseous carbon oxides that contribute to increased porosity (**Wan & Wang, 2018**). Therefore, oxidation processes under air or oxygen-rich environments can substantially modify pore morphology, interparticle bonding, and phase composition. In contrast, in inert atmospheres such as argon, oxidation is suppressed, and the resulting microstructure depends more on viscous flow and particle rearrangement. These mechanisms yield distinct structural features that directly influence both thermal conductivity and mechanical strength (**Chen &Miyamoto, 2014**).

The relationship between porosity and thermal transport in ceramics is well-documented. The introduction of pores disrupts the phonon transport path, reducing the effective thermal conductivity due to enhanced phonon scattering at solid-pore interfaces (**Chun et al., 2005**). However, excessive porosity can severely weaken mechanical strength, as the load-bearing cross-section is reduced and stress concentration arises around pores (**Kim et al., 2020, Wan & Wang, 2018**). Thus, an optimal microstructure must balance porosity for insulation and sufficient strength for mechanical stability. The addition of nano-SiC particles is particularly interesting because nano-sized SiC can alter sintering behavior, influence pore evolution, and improve interfacial bonding at moderate temperatures (**Su et al., 2018**). At the same time, nanoscale SiC may also promote heterogeneous oxidation and interfacial phonon scattering, leading to additional reductions in thermal conductivity (**Streitwieser et al., 2005**). Annealing, or controlled post-sintering heat treatment, is a powerful technique to modify ceramic microstructure without significant changes in composition. The annealing atmosphere is especially critical: an inert gas such as argon limits oxidation and helps preserve SiC integrity, while an oxidizing atmosphere (air) induces surface oxidation, forming SiO_2-rich layers that can seal pores or, alternatively, generate new ones depending on the oxidation kinetics.

Studies have demonstrated that annealing at intermediate temperatures (900–1200 °C) under air results in the gradual oxidation of SiC, generating amorphous SiO_2 films that may enhance the overall insulating capability but reduce strength due to interfacial debonding **(Malik et al., 2020)**. In contrast, annealing under argon tends to stabilize the as-formed microstructure and preserve mechanical properties. Although individual effects of oxidation and porosity on SiC and SiO_2 ceramics have been widely reported, comparative studies analyzing the interplay of annealing atmosphere and nano-SiC content in SiO_2–SiC composites remain limited. Recent reports on hierarchical porous ceramics have suggested that the combined use of nano-sized reinforcements and tailored annealing conditions can yield materials with both ultralow thermal conductivity (< 0.06 W m^{-1} K^{-1}) and acceptable compressive strength (> 10 MPa) **(Jana et al., 2017)**. However, achieving this balance requires a clear understanding of how pore morphology, oxidation reactions, and interfacial phenomena evolve under different atmospheres. Furthermore, the growing demand for lightweight insulation materials in aerospace, energy, and environmental technologies underscores the need for ceramics that combine low thermal conductivity with sufficient mechanical robustness. For instance, in gas turbines and combustion systems, thermal barriers must resist temperatures beyond 1000 °C while maintaining dimensional stability. In energy storage and conversion systems, such as solid oxide fuel cells, materials with tailored porosity are needed to minimize thermal losses while ensuring structural integrity. The SiO_2–SiC system provides an adaptable platform for these applications, but systematic studies under well-controlled annealing conditions are required to optimize its potential.

Therefore, the present study investigates the effect of annealing at 1000 °C for 1 h in argon and air atmospheres on the porous SiO_2–SiC ceramics with varying nano-SiC contents (0–35 wt%). The focus is on elucidating the interrelation between porosity, microstructure, thermal conductivity, and compressive strength. By comparing inert and oxidizing annealing environments, this work aims to reveal the mechanisms that govern microstructural evolution and property variations in SiO_2–SiC composites. The findings will contribute to the rational design of low-thermal-conductivity porous ceramics with optimized performance for high-temperature insulation and energy applications.

2. LITERATURE REVIEW

Porous SiC and SiC-based composite ceramics have been studied extensively. **Malik et al., (2020)** observed that both thermal conductivity and mechanical strength tend to decrease as porosity increases, though particle size and intergranular bonding can modulate this trend. **Kang et al. (2021)** reported extremely low thermal conductivities in porous nano-SiC via multiple thermal resistance engineering.

In the specific SiO_2–SiC system, oxidation of SiC to SiO_2 can act as a sintering aid, modifying microstructural connectivity and pore geometry **(Gomez-Gomez et al., 2019).** The interfacial thermal boundary resistance between SiO_2 and SiC phases can further suppress phonon transport **(Manoji Kumar et al., 2011)**. Recent work on hierarchical porous ceramics combining SiC and oxide phases has shown promising insulation properties.

However, there is a lack of studies focusing specifically on how annealing atmosphere (Ar vs. air) at moderate temperatures (e.g. 1000 °C) influences the tradeoff between thermal insulation and mechanical strength in SiO_2–SiC composites. This gap motivates the present work.

3. MATERIALS AND METHODS

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 800 °C for 2 h, then subsequently annealed at 1000 °C for 1 h in air and argon atmosphere.

The sintered specimens were designated as follows: for example, SA-10 and SAr-10, where the number after the dash (10) indicates the sintering temperature of 1000 °C, A and Ar means air and argon atmosphere, respectively.

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

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

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:

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

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

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

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

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

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]:

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

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

4 RESULTS AND DISCUSSION

4.1 Porosity and Microstructure

Figure 1 shows the porosity of the porous SiO_2-SiC based ceramics as a function of initial nano-SiC content. With increasing SiC content, porosity of argon-annealed samples increased from 75.2 % to 76.0 %, while for air-annealed samples it increased from 64.8 % to 67.5 %. The porosity of argon-annealed porous SiO_2-SiC based ceramics were 75.2% for SAr0-10, 75.7% for SAr10-10, 75.8% for SAr20-10, and 76.0% for SAr35-10.

In argon atmosphere, the increase is modest; presence of SiC particles impedes viscous flow of SiO_2 and densification, thus stabilizing the pore network. In air, oxidation of SiC leads to the formation of additional SiO_2 and gaseous CO/CO_2, which expands the pore volume and introduces new pore channels, thus boosting porosity more noticeably.

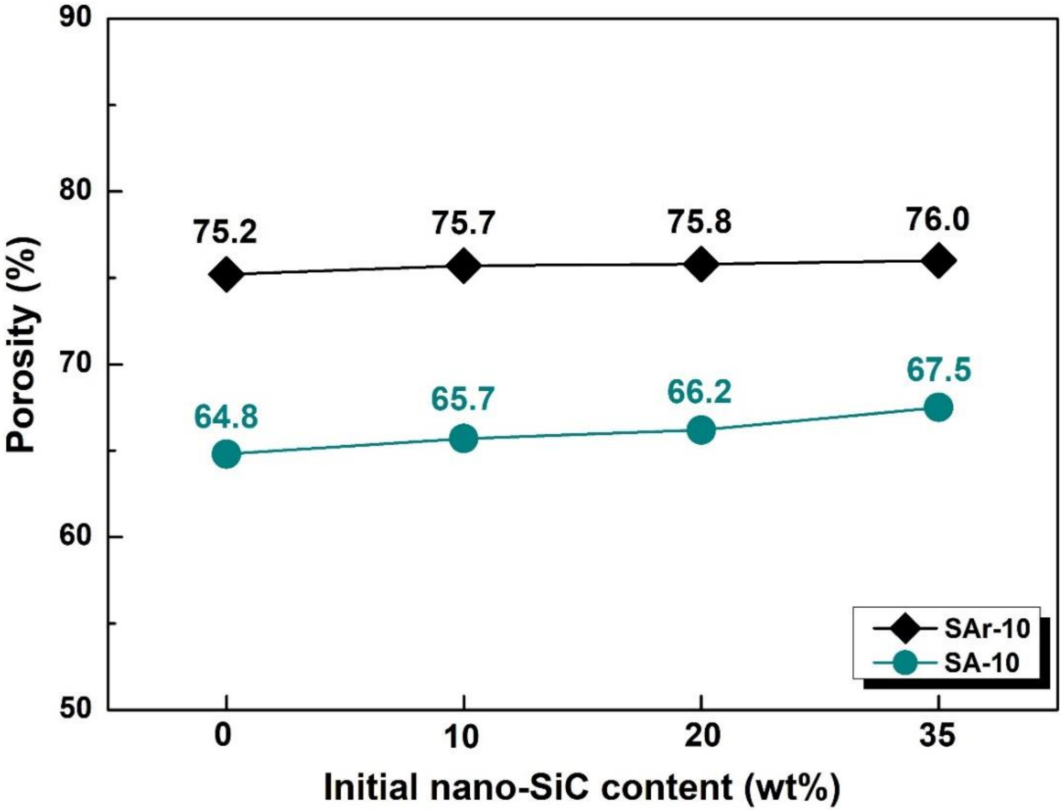

Figure 1 – Porosity of the porous SiO_2-SiC based ceramics as a function of initial nano-SiC content.

4.2 Thermal conductivity

The thermal Conductivity of the porous SiO_2-SiC based ceramics as a function of initial nano-SiC content has shown in **Figure 2**. Porous SiO_2-SiC based ceramics annealed in argon atmosphere showed the decrease in thermal conductivity from 0.152 – 0.122 W m^{-1} K^{-1} when initial nano-SiC content increased from 0 to 35 wt%. The thermal conductivity of air-annealed porous SiC ceramics decreased from 0.058 → 0.045 W m^{-1} K^{-1} as initial nano-SiC content increases from 0 to 35 wt%.

The marked decrease of thermal conductivity correlates with increased porosity and disruption in continuous conduction paths. The air-annealed samples show much lower conductivity, reflecting both the higher porosity and enhanced phonon scattering at interfaces, including possible amorphous SiO_2 layers formed via oxidation. The role of thermal boundary resistance between SiO_2 and SiC phases becomes more significant in these mixed-phase porous systems.

Compared to literature, the conductivity range is competitive with other low-k porous systems (**e.g., Kang et al. 2021**, 0.14 W m^{-1} K^{-1}) or the composite SiC–SiO_2–Al_2O_3–TiO_2 system achieving ~0.059 W m^{-1} K^{-1} at ~74% porosity.

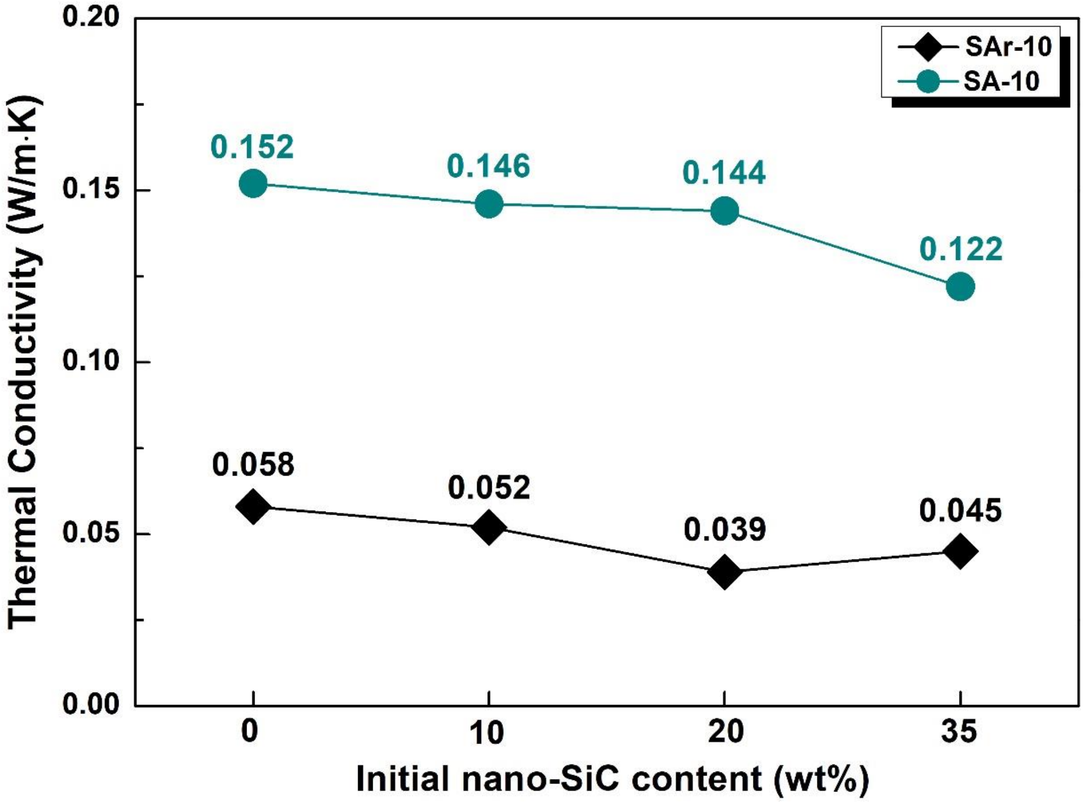

Figure 2 – Thermal Conductivity of the porous SiO_2-SiC based ceramics as a function of initial nano-SiC content.

4.3 Compressive strength

Compressive strengths decrease as initial nano-SiC content increases **(Figure 3)**. Porous SiO_2-SiC based ceramics annealed in argon atmosphere showed the decrease in compressive strength from 24.8 – 14.1 MPa when initial nano-SiC content increased from 0 to 35 wt%. The compressive strength of air annealed porous SiC ceramics decreased from 10.6 – 4.0 MPa as initial nano-SiC content increases from 0 to 35 wt%.

The decline in strength is directly linked to increased porosity, reduced cross-sectional load-bearing area, and more defects or pore stress concentrators. In air-annealed samples, the oxidation-driven creation of weak SiO_2 phases and larger pores exacerbates mechanical degradation. The argon-annealed samples maintain relatively higher strength because of limited oxidation and more coherent phase interfaces.

In a log–porosity plot, one might expect a linear or near-linear relation consistent with empirical strength–porosity models. The stronger performance under argon following the same porosity range suggests that microstructural control (e.g. pore geometry, neck thickness) is better preserved in inert atmosphere.

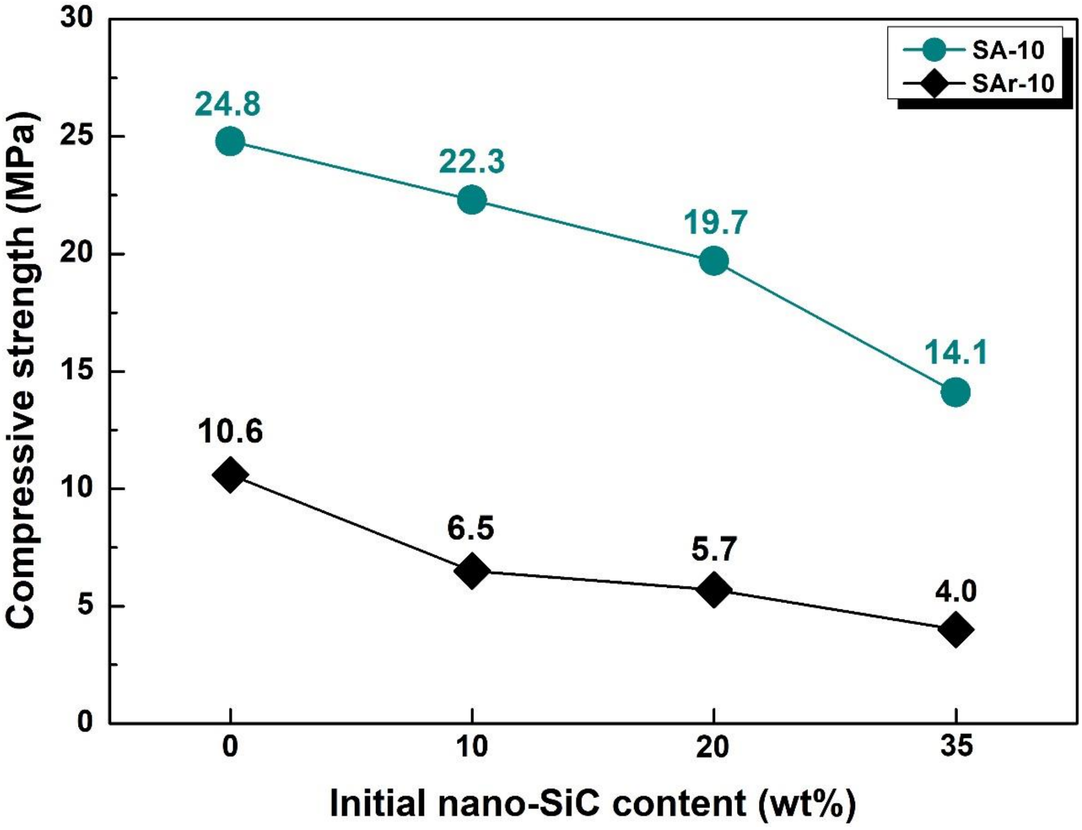

Figure 3 – Compressive strength of the porous SiO_2-SiC based ceramics as a function of initial nano-SiC content.

5 CONCLUSIONS

The effects of annealing atmosphere (argon vs. air) at 1000 °C for 1 h on porous SiO_2–SiC ceramics with varying nano-SiC contents were systematically studied. Key findings:

(1) Porosity increases with SiC content in both atmospheres; the increase is more substantial in air due to oxidation-induced pore formation.

(2) Thermal conductivity decreases correspondingly, with the lowest value of 0.045 W m^{-1} K^{-1} achieved in the air series.

(3) Compressive strength diminishes with SiC addition, with argon-annealed samples retaining higher strength compared to air-annealed ones.

These results illustrate that annealing atmosphere is a crucial parameter in balancing thermal insulation performance and mechanical reliability in porous SiO_2–SiC ceramics. Annealing in air yields extremely low thermal conductivity but at the cost of mechanical strength, while argon annealing provides a more favorable balance for structural applications.

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