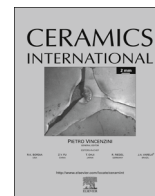




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Influence of silicon carbide as a mineralizer on mechanical and thermal properties of silica-based ceramic cores

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ABSTRACT

Ceramic cores have been designed with compounds based on fused silica due to its excellent thermal stability and chemical inertness against molten metals. To endure the high temperatures present during investment casting, mineralizers have been widely used to enhance the flexural strength and shrinkage of ceramic cores. In this study, we demonstrated a silica-based ceramic core with silicon carbide as a mineralizer for improving the mechanical and thermal properties. The SiC in the silica-based ceramic cores can enhance the mechanical properties (i.e., flexural strength and linear shrinkage) by playing a role as a seed for the crystallization of fused silica to cristobalite. The SiC also improves the thermal conductivity due to its higher value compared with fused silica. The results suggest that using the optimal amount of silicon carbide in silica-based ceramic cores can provide excellent mechanical properties of flexural strength and linear shrinkage and improved thermal conductivity.

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1. Introduction

Fused silica-based ceramic cores have been used to manufacture high-temperature gas turbine parts (e.g., blades and vanes) with hollow structures [1,2]. Intricate core bodies for turbine blades are produced by ceramic injection molding, followed by high-temperature firing. The conventional properties of cores include dimensional stability and a certain degree of strength, for example, a sintering shrinkage as low as 1% and a flexural strength of 10–40 MPa that can endure the high thermal load applied in the several steps of the mold-making process: wax casting, shell firing and subsequent investment casting of the superalloy [3,4].

Fused silica is conventionally used due to its low coefficient of thermal expansion ($0.55 \times 10^{-6}/\text{K}$, between 25 °C and 1000 °C), excellent chemical stability with molten metals, and good leachability [5]. Nonetheless, a silica-based core does not exhibit low-strength firing without the use of additives or sintering aids. The fracture strength of fused silica for core application has been reported to be not less than 5–6 MPa [6], depending on the powder batch, purity, particle diameter, and other parameters. The ceramic powder batch is composed of several powders ranging from millimeter-sized to sub-micron-sized. In addition, the powder batch is composed of mainly coarse powders that are not well sintered.

The usage of various sizes of powder is required for easily removal but makes it difficult to obtain a mechanically unfractured body. Another approach for the enhancement of the flexural strength is addressed by the addition of a mineralizer, which assists in the crystallization of the fused silica [2].

On the other hand, the dimensional change of silica-based ceramic cores during the investment casting of gas turbine hot parts should be considered, as it results in the dimensional inaccuracy of the final cast product. To melt the superalloys, investment casting is usually carried out at high temperature. This can cause the core body to be significantly shrunk, preventing the introduction of the final product due to incorrect dimensions. Lequeux et al. reported that boehmite infiltration reduced the contraction of silica-based cores [7]. The formation of aluminosilicate via an alumina precursor (colloidal boehmite) on silica grains inhibited the viscous flow of fused silica, reducing the densification shrinkage. In this study, we demonstrated a silica-based core with added silicon carbide (SiC) to enhance the mechanical properties and size stability. SiC has a high flexural strength of 588 MPa and thus can improve the strength of silica-based composites [8,9]. SiC, which has a crystalline phase, can reduce the shrinkage of silica-based composites by preventing the viscous flow of fused silica for densification. In addition, SiC can increase the thermal conductivity of silica-based composites due to its high thermal conductivity ($k_{\text{SiC}}=490 \text{ W/m K}$) compared with that of fused silica of $1.3 \text{ W/m}\cdot\text{K}$ at 20 °C [10].

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2. Materials and methods

The mixture for the fabrication of the ceramic core consisted of fused silica (99.6% purity, Boram Chemetal, Korea) with various particle sizes ranging from 25 to 150 μm , zircon flour (97.5% purity, 1 μm , Cenotec, Korea) and SiC (97.5%, 400mesh, Sigma-Aldrich, United States). All ceramic powders are commercially available. The fused silica and zircon were used at a ratio of 3:1 as a compound for the silica-based composites. The SiC contents of the ceramic powders were 5, 10, 15 and 20 wt%. To prepare a flowable slurry for injection molding, the mixture was mixed with 15 wt% of thermoplastic binders: paraffin wax (M.P. 69 °C, Nippon-Seiro, Japan), microcrystalline wax (M.P. 82 °C, Nippon-Seiro, Japan), oleic acid ($\text{C}_{19}\text{H}_{34}\text{O}_2$, Samchun Pure Chemical, Korea), and stearic acid ($\text{C}_{19}\text{H}_{36}\text{O}_2$, Samchun Pure Chemical, Korea). The ceramic powders were ball-milled for 6 h at room temperature with zirconia balls. To prepare the feedstocks for injection molding, the ball-milled ceramic powders were mixed with melted thermoplastic binders for 6 h at 80 °C under vacuum. The mixed feedstocks were fed into a storage chamber of a C-frame ceramic injection molding machine (CTM-CI-CF-35, Cleveland Tools and Machines, OH, United States) and injected at a temperature of 80 °C, an injection pressure of 60 bar and a flow rate of 400 cc/sec. The dimensions of the specimens used for measuring the flexural strength were 6 × 8 × 90 mm (W × L × H). The injected specimens were heat-treated at 1200 °C for 2 h at a heating rate of 5 K/min using a chamber furnace (UAF-15-27-LHE, Lenton, England). During the heat treatment, the specimens were positioned in a crucible with backfill powder to prevent collapse caused by the melting of the thermoplastic binders. A dilatometer (DIL 402C, Netzsch, Germany) was used to measure the thermal expansion and contraction of the specimens. The thermal diffusivity of pelletized samples with a diameter of 12.7 mm and a thickness of 1.5 mm was evaluated using a laser-flash analyzer (LFA, LFA-427, Netzsch, Germany) under an argon atmosphere in the temperature range of 25 to 1300 °C. Before analysis, the surfaces of the pellet specimens were coated with carbon spray to prevent laser transmission. The thermal conductivity (k) of the specimens is the product of the apparent density (ρ), specific heat (C_p) and the measured thermal diffusivity (λ). For the calculation of the thermal conductivity, we used density and specific heat data at the elevated temperature supplied by the equipment manufacturer.

The phase transformations of the specimens were detected with an X-ray diffractometer (XRD, D/Max-2200, Rigaku, Japan) with Cu K α radiation ($\lambda_{\text{CuK}\alpha} = 1.54178 \text{ \AA}$). The flexural strength testing was performed with a 3-point bending fixture with a span of 80 mm on a universal testing machine (UTM, H10SK, Hounsfield, England) at a crosshead speed of 1 mm/min according to ASTM C 1161-13. Five specimens were tested for each value. The microstructures of the specimens were observed via field-emission scanning electron microscopy (FE-SEM, S-4800, Hitachi, Japan). The apparent density and porosity of the specimens were determined based on Archimedes' method. To examine the SiC oxidation in the specimens, a powder mixture without thermoplastic binders was analyzed using a thermo-gravimetric analyzer (TGA, SDT 2960 Simultaneous, TA Instruments, Delaware, United States) at 1200 °C with a heating rate of 5 K/min. The leaching test was carried out using an autoclave with 40% sodium hydroxide (NaOH) solution. 10 g of specimen was added into 100 mL of solution and leached at 150 °C for 1 h with an atmospheric pressure of 4 bars.

3. Results and discussion

X-ray diffraction patterns of the silica-based composites are shown in Fig. 1. The specimen without SiC (K0) shows only zircon

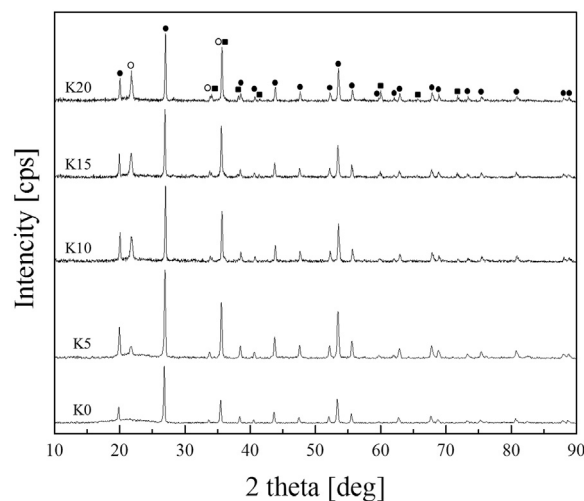


Fig. 1. XRD patterns of specimens with various contents of SiC; ●: zircon, ○: cristobalite and ■: silicon carbide.

diffraction peaks (JCPDS 6-266). The fused silica was not detected on the XRD pattern due to its amorphous structure. SiC peaks corresponding to JCPDS 73-1664 were observed in the XRD patterns when the specimen contained more than 10 wt% SiC. Cristobalite peaks (JCPDS 39-1425) were observed in all specimens that contained SiC (K5, 10, 15 and 20), and their intensity gradually increased with the increasing content. Table 1 shows the cristobalite peak ($2\theta = 21.86^\circ$, (110) plane) to zircon peak ($2\theta = 26.86^\circ$, (200) plane) ratio of the specimens. Because zircon does not phase-transform at less than 1600 °C, it is possible to estimate the relative content of cristobalite in silica-zircon composites by comparison of the cristobalite and zircon peaks [11–13]. The peak ratio significantly increased with the amount of SiC. The peak ratios from the XRD patterns indicate that SiC promotes the formation of cristobalite, and its relative content increased with the SiC addition. It is well known that fused silica crystallizes to β -cristobalite at a high temperature of approximately 1300 °C during the heating segment [1,2]. Impurities in the raw silica, such as alumina or cristobalite, can accelerate the development of cristobalite by acting as a seed for the crystallization of the fused silica [7,12,14]. The XRD results demonstrated that the crystallization of fused silica to cristobalite was facilitated in the presence of SiC. During heat treatment in air conditions, the surface of SiC oxidizes to amorphous SiO_2 , and it further changes to cristobalite via crystallization (or devitrification) at 1200 °C [15]. However, the cristobalite peaks were not revealed in the XRD pattern of SiC heated at 1200 °C for 2 h because only a small amount was generated on the surface of the SiC. Calculating the oxidation rate is necessary because the oxidation of SiC can affect the mechanical and thermal properties of the specimens. Table 2 shows the calculated oxidation fraction of the SiC-containing specimens. The weight gain measured by thermo-gravimetric analysis and the SiC content in the batch were used for calculation. TGA was performed at 1200 °C for 2 h with a heating rate of 5 K/min. The sintered

Table 1
The peak ratio of cristobalite to zircon of silica-based composites with various contents of SiC.

SiC content	Cristobalite/zircon peak ratio
0 wt%	0.067
5 wt%	0.137
10 wt%	0.341
15 wt%	0.367
20 wt%	0.457

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