



Contents lists available at ScienceDirect

Ceramics International

journal homepage: www.elsevier.com/locate/ceramint

Improvement of the mechanical properties of SiC reticulated porous ceramics with optimized three-layered struts for porous media combustion

Xiong Liang^a, Yawei Li^{a,*}, Jun Liu^a, Shaobai Sang^a, Yuanyuan Chen^a, Benwen Li^b,
Christos G. Aneziris^c

^a The State Key Laboratory of Refractories and Metallurgy, Wuhan University of Science and Technology, Wuhan 430081, China

^b Institute of Thermal Engineering, School of Energy and Power Engineering, Dalian University of Technology, Dalian 116024, China

^c Technical University Bergakademie Freiberg, Institute for Ceramic, Glass and Construction Materials, Agricolastraße 17, 09596 Freiberg, Germany

ARTICLE INFO

Keywords:

SiC reticulated porous ceramics
Reaction sintering
Mechanical properties
Thermal shock resistance

ABSTRACT

Silicon carbide reticulated porous ceramics (SiC RPCs) with three-layered struts were fabricated by polymer replica method, followed by infiltrating alumina slurries containing silicon (slurry-Si) and andalusite (slurry-An), respectively. The effects of composition of infiltration slurries on the strut structure, mechanical properties and thermal shock resistance of SiC RPCs were investigated. The results showed that the SiC RPCs infiltrated with slurry-Si and slurry-An exhibited better mechanical properties and thermal shock resistance in comparison with those of alumina slurry infiltration, even obtained the considerable strength at 1300 °C. In slurry-Si, silicon was oxidized into SiO₂ in the temperature range from 1300 °C to 1400 °C and it reacted with Al₂O₃ into mullite phase at 1450 °C. Meantime, the addition of silicon in slurry-Si could reduce SiC oxidation of SiC RPCs during firing process in contrast with alumina slurry. With regard to slurry-An, andalusite started to transform into mullite phase at 1300 °C and the secondary mullitization occurred at 1450 °C. The enhanced mechanical properties and thermal shock resistance of SiC RPCs infiltrated alumina slurries containing silicon and andalusite were attributed to the optimized microstructure and the triangular zone (inner layer of strut) with mullite bonded corundum via reaction sintering. In addition, the generation of residual compressive stress together with better interlocked needle-like mullite led to the crack-deflection in SiC skeleton, thus improving the thermal shock resistance of obtained SiC RPCs.

1. Introduction

Due to the advantages of porous media combustion of premixed gases, such as low emission of NO_x and CO, high power density, large range of modulation, etc., it can be applied in internal combustion engines, low calorific gas burners, volatile organic compound oxidizers and radiation heaters [1,2]. Reticulated porous ceramics (RPCs) as one of the burners for porous media combustion (PMC) are characterized by an open, three-dimensional network structure [3]. However, the scour of flue gas and large thermal stress from the sharp temperature gradients in service made porous burners easily damaged. Therefore, the mechanical properties and thermal shock resistance of RPCs were necessary to be enhanced for the longevity of burners [4,5]. In this case, silicon carbide RPCs (SiC RPCs) was selected as candidate material for porous components of PMC because of its lower thermal expansion coefficient, high thermal conductivity and strength [6].

One of the most popular methods to prepare RPCs was a replication

process, more specifically defined as the polymeric sponge one [7]. Generally, RPCs produced in this method had low strength and fracture toughness because of the ceramic skeleton with hollow struts and large flaws after the polymer template was burned out, which made them sensitive to structural stresses and limited long term service [8]. So, some ways, e.g. pretreatment of polymeric sponge and second slurry coating or vacuum infiltration, were adopted to improve the mechanical properties of RPCs. Yao et al. [9] increased the surface roughness and wetting ability of polymeric sponge by the treatment of acid/alkali or polycarbosilane. The modified sponge was beneficial to thicken the ceramic skeleton. Jun et al. [10] coated carbon black slurry on the polymeric sponge before the immersion process of ceramic slurry with an aim to blunt the sharp apices of triangular voids and reduce the stress concentration in the ceramic strut. Furthermore, Zhu and Pu et al. [11,12] strengthened RPCs through recoating technique. The surface flaws in struts were eliminated and struts were greatly thickened after the second coating process. Vogt et al. [13] filled

* Corresponding author.

E-mail address: liyawei@wust.edu.cn (Y. Li).

<http://dx.doi.org/10.1016/j.ceramint.2016.12.007>

Received 19 August 2016; Received in revised form 24 November 2016; Accepted 2 December 2016
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Table 1
Compositions of the vacuum infiltration slurries.

Infiltration slurry	α -Al ₂ O ₃	Silicon	Andalusite	polycarboxylate FS	Contraspum K 1012	Solid content wt%
Slurry-A	100	–	–	0.3	0.1	77.0
Slurry-Si	95	5	–	0.3	0.1	77.0
Slurry-An	74	–	26	0.3	0.1	77.0

alumina slurry into triangular voids of the struts via vacuum infiltration process, the dense struts resulted in the improved strength of alumina RPCs.

With regard to the ceramic laminates, they exhibited excellent damage resistance and fracture toughness because of the multi-layered structure of composites. In general, these multi-layered laminates were designed to consist of one or more compressive layers [14,15]. It had been reported that the presence of compressive stresses could effectively inhibit or delay the occurrence and prorogation of cracks in ceramic laminates, and therefore significantly increase the damage resistance and thermal shock resistance [16]. Ceramic laminates usually possessed multi-layers with well-bonded interface via reaction sintering. In this case, the residual compressive stresses could be generated either by a thermal expansion mismatch between the multi-layers or by a volume expansion of a dispersed phase in one of the layers due to phase transformations [17].

In this work, SiC RPCs with multi-layered struts was fabricated by polymer replica technique, followed by vacuum infiltration of alumina slurry containing silica source. Silicon and andalusite powders were added into the infiltrated alumina slurry to in-situ form mullite via reaction sintering and optimize the three-layered silicon carbide-based struts. On the one hand, the reaction sintering of silicon and alumina will increase the strength of SiC RPCs, in the meantime the silicon is added to decrease the oxidization of SiC during heating-up because it is oxidized prior to SiC. On the other hand, andalusite itself with a feature of good thermal shock resistance might endow SiC RPCs with better mechanical properties and thermal shock resistance. Meanwhile, the reaction sintering of silica source and alumina in infiltration slurry will make the thermal expansion mismatch between SiC skeleton and outer layer, thus resulting in the generation of residual compressive stresses in SiC skeleton during cooling from sintering temperature. In this experiment, the effects of the addition of silicon and andalusite in the infiltration slurry on microstructure and mechanical properties of SiC RPCs were investigated, respectively. Following that the thermal shock behavior of RPCs fired at 1450 °C was also discussed by the strength change and crack propagation on the struts before and after thermal cycles.

2. Experimental

2.1. Preparation of the specimens

The raw materials used for preparing silicon carbide coating slurry were commercial available SiC powder (< 45 μ m, 98%, China), α -Al₂O₃ (~1.2 μ m, Henan Special Refractories Co., Ltd., China) and microsilica powder (~0.5 μ m, 951UL, Elkem, Norway). The weight percentage of SiC powder was 80 wt%, α -Al₂O₃ and microsilica were 14.16 wt% and 5.84 wt%, respectively. The additives were polycarboxylate (FS, BASF Group, Germany—used as a dispersant agent), ammonium lignosulfonate (Tianjin institute of fines chemicals, China, referred to as AL hereafter—used as binder), sodium carboxymethyl-cellulose (CMC, Sinopharm Chemical Reagent Co., Ltd, China—used as a thickening agent), and contraspum K 1012 (Zschimmer & Schwarz, Lahnstein, Germany—used as an antifoam agent). The weight percentages of FS, AL, CMC and K1012 based on the ceramic powder were 0.3 wt%, 1.5 wt%, 0.3 wt% and 0.1 wt% in silicon carbide coating slurry, respectively. The SiC slurry with initial solid content of 77.4 wt% were prepared by

mixing SiC powder, α -Al₂O₃ and microsilica with additives (FS, AL, CMC and K1012) in the deionized water. After stirred for 1 h at a rotate speed of 300 r/min, the SiC slurry with a high thixotropic could be obtained. The polyurethane open-cell sponge template (10 pores/inch, F. M Co. Ltd., Germany) with dimensions of 50×50×20 mm³ were immersed into the as-prepared silicon carbide slurry, followed by passing through preset roller to remove excess slurry. Remaining closed pores were eliminated by blowing carefully compressed air through the sponge structure. After drying, the coated sponges were heated to 850 °C to produce SiC reticulated preforms with good handling strength.

In vacuum infiltration process, the above α -Al₂O₃ powder was the main material for preparing infiltration slurries. Silicon powder (Si, < 3 μ m, 98%, China), andalusite (d₅₀=7.1 μ m, Al₂O₃=59.78 wt%, Y60, Imerys) were used as silica source in infiltration slurries, respectively. The composition of infiltration slurries was listed in Table 1. It was designed that the molar ratio of Al₂O₃/SiO₂ (based on the oxidation of silicon) was 5.2 both in slurry-Si and slurry-An. Deionized water was firstly mixed with FS and K 1012 by stirring for about 5 min. Subsequently the alumina mixture powders with or without silica source were added into the solution and milled for 3 h using alumina balls. Then SiC preforms were totally immersed into the as-prepared infiltration slurries and a vacuum of 0.5 Pa was applied for 20 min. After the as-infiltrated SiC preforms were dried at 110 °C, they were fired in air at 1300 °C, 1400 °C and 1450 °C for 3 h, respectively. The infiltrated preforms with slurry-A, slurry-Si and slurry-An were named as SA, SS and SAn, respectively. Finally, the SiC coating slurry and infiltration slurries were respectively casted in a mold of 25 mm×25 mm×125 mm. After being placed at room temperature for 24 h, these molds were dried at 110 °C. The firing regime of the rectangular bars were carried out according to the SiC RPCs specimens.

2.2. Characterization

The RPCs density ρ_{RPC} was the density of the whole specimen and was calculated with the equation:

$$\rho_{\text{RPC}} = \frac{m}{l \cdot b \cdot h}$$

where l was the length, b the width, h the height, and m the mass of the RPCs specimen.

The thermal expansion coefficient of SiC slurry and infiltration slurries were evaluated by a dilatometer DIL 402 C (Netzsch, Selb, Germany) on the slip casted cylinder samples (Φ 8 mm×50 mm). The Young's modulus of these slurries (casted as rectangular bars of 20 mm×20 mm×120 mm) were measured by the impulse excitation technique with the RFDA (IMCE, Genk, Belgium) at room temperature. The macrostructure of RPCs was characterized by digital camera and the diameter of struts were analyzed by Image-Pro plus software (Media Cybernetics, Inc., Netherlands). SiC oxidization ratio was calculated by mass change before and after firing process to evaluate the oxidation degree of SiC in RPCs. Mechanical tests on the fired RPCs were performed by determining the cold compressive strength (CCS) with a universal testing machine (ETM, Wance, China), using a load speed of 0.5 mm/min. Specimens of the size 50 mm×50 mm×20 mm were positioned between loading plates (Φ 100×20 mm). A cardboard of 4 mm thickness was placed between loading plates and specimens to obtain a uniform loading. The thermal shock resistance of SiC RPCs

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