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Effect of recoating slurry compositions on the microstructure and properties of SiC reticulated porous ceramics

Xiumin Yao *, Yong Yang, Xuejian Liu, Zhengren Huang

Structural Ceramics Engineering Research Center, Shanghai Institute of Ceramics, Chinese Academy of Sciences, 1295 Dingxi Road, Shanghai 200050, PR China

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Abstract

Silicon carbide reticulated porous ceramics (SiC RPCs) were fabricated by polymer sponge replica technique, followed by recoating with SiC slurries of two different sintering additives of $MgO–Al_2O_3–SiO_2$ (Slurry 1) and polycarbosilane (Slurry 2). The sintering temperature of SiC RPCs recoated with Slurry 2 was 1100 °C, which was 200 °C lower than that for one recoated with Slurry 1. The prepared SiC RPCs exhibited homogeneous microstructure and contained pores with different sizes which was entrapped in the strut of SiC RPCs, small pores with diameter lower than 4 μm and large pores with diameter higher than 10 μm. Bending strength of SiC RPCs recoated with Slurry 1 was two times higher than that for the non-recoated samples, which was 1.88 MPa and was a little higher than that for one recoated with slurry 2. At the same time, high thermal shock resistance and high refractoriness were achieved for SiC RPCs recoated with Slurry 2.

Keywords: SiC reticulated porous ceramic; Recoating slurry compositions; Compressive strength; Thermal shock resistance

1. Introduction

Reticulated porous ceramics (RPCs) with an open and three-dimensional network structure are widely applied in the field of high temperature materials such as molten metal filters, diesel engine exhaust filters and catalyst supports due to their high permeability, resistance to chemical attack and structural uniformity.[1–4] In particular, silicon carbide RPCs (SiC RPCs) is one of the best candidate materials for high temperature filter because of its lower thermal expansion coefficient, high thermal conductivity and high strength.[5] The currently manufacturing method of these RPCs is the polymer sponge replica technique.[6] In this method, green RPCs are prepared to contain very few filled cells by coating a polyurethane sponge with a thixotropic ceramic slurry. After drying the green body to eliminate the polymer template, they are sintered at high temperature to form a porous ceramics. Many kinds of SiC slurries have been developed to contain $Al_2O_3–SiO_2$[6–9] or $MgO–Al_2O_3–SiO_2$[10,11] as the sintering additives, the effect of which on the structure and properties of SiC RPCs are reported. Further, an innovative method has been proposed to fabricate the SiC ceramic foams using preceramic polymers as the precursor of silicon carbide. Bao et al.[12,13] immersed polyurethane foams in polysilane precursor solutions to prepare pre-foams. The prepared pre-foams were subjected to firing at high temperature, forming final ceramic foams. Fitzgerald et al.[14] infiltrated a preceramic precursor solution in a porous substrate, which was removed by chemical leaching to leave an open and interconnected porous ceramics. However, the prepared SiC foams exhibited low mechanical strength, which limit their use to the structural applications.[12–14] To improve the mechanical properties of the SiC RPCs, the slurry coverage has been testified.[15–19] A second slurry coating[7,20] or a vacuum infiltration process[21] of the foams was applied to fill any flaws in the first coating layer.

In this paper, we applied the recoating technique to form the SiC RPCs and investigated the effect of the slurry compositions on the microstructure and mechanical properties of the obtained SiC PRCs. The SiC preforms were prepared by polymer sponge replica technique using preset roller technique, followed by coating the slurries with two different compositions by a centrifugal process. The SiC performs, prepared by polymer sponge replica technique, exhibit higher strength than those prepared by preceramic polymer precursor's methods, which is advantage to the following recoating process. The effect of composition of the

* Corresponding author. Tel.: +86 21 5241 4224; fax: +86 21 5241 3903.
E-mail address: xmyao@mail.sic.ac.cn (X. Yao).

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recoating slurry was discussed to relate with the microstructure and properties of SiC RPCs ceramics.

2. Experimental procedure

2.1. Preparation of SiC green bodies

The green bodies of SiC RPCs with $MgO–Al_2O_3–SiO_2$ as sintering additive system were prepared by polymer sponge replica technique. Alumina powders, talc particles and kaolin powders were added as $MgO–Al_2O_3–SiO_2$ sintering aids.

The SiC slurries (80 wt% solid contents) for the green bodies were prepared by the following procedure. Deionized water was first mixed with silica sol by stirring for about 10 min in an attritor (Szegvari Attritor System 01HD, Union process Inc., USA) using alumina balls as grinding media. Ceramic powders were subsequently added to the solution and stirred for 3 h. Then, a thickening agent (sodium carboxymethyl-cellulose) and an antifoaming agent (SAG 630, Witco Hongkong Limited Co., China) were added to the slurry and kept stirring for another 3 h.

The polyurethane sponges were immersed in the above slurry. Then the sponges with slurry were passed by a preset roller to remove excess slurry. After being dried, the coated sponge substrates were heated at a heating rate of 1 °C/min to burn out the sponge at 600 °C, and then heated at 900 °C during 3 h with a hating rate of 5 °C/min. The detail of the process is reported in our previous article.[11]

2.2. Preparation slurries for recoating

In this study, we adopted two different coating slurries contain $MgO–Al_2O_3–SiO_2$ ceramic powder and polycarbosilane (PCS), which are thereafter denoted as Slurry 1 and Slurry 2, respectively. The Slurry1 (70 wt% solid contents) was prepared by the same procedure as the green body preparation.

For the preparation of Slurry 2, 15 wt% PCS (average molecule weight ~1250, National University of Defense Technology, China) was mixed with gasoline by ball milling using alumina balls for about 1 h. SiC powders were subsequently added to the solution, followed by milling for 3 h.

2.3. Samples preparation

In the preset roller method, the polyurethane sponges were immersed in high thixotropic SiC slurry containing $MgO–Al_2O_3–SiO_2$ as sintering aids, followed by passing through preset roller to remove excess slurry. After being dried, the coated sponge substrates were heated in air up to 600 °C at a heating rate of 1 °C/min to burn out the sponge, and then heated to 900 °C at a heating rate of 5 °C/min and kept for 1 h at that temperature to produce reticulated preforms with handling strength.

In the recoating process, the obtained preforms were coated repeatedly with the prepared slurries using centrifugal process to form the thinner layers. After being dried, the preforms recoated with Slurry 1 were sintered in air at 1300 °C for 3 h at a heating rate of 5 °C/min. When the preforms were recoated with Slurry 2, they were heated at 280 °C for 0.5 h to make PCS mutual crosslinking, followed by heating at a heating rate of 1 °C/min to 1000–1300°C under N_2 atmosphere, and then kept for 1 h at the predetermined temperature.

2.4. Measurements of properties

The macrostructure of RPCs was characterized by digital camera (Olympus C-5050, Olympus Optical Co., Ltd., Japan) and the microstructure of struts was observed by scanning electron microscopy (SEM) (model EPMA-8705Q and JAX-8100, Japan). Bulk density of RPCs, ρ_b, was determined from the dimensions and mass of the sintered samples. The apparent density (ρ_s) and open porosity of the struts were measured using mercury porosimetry (Model Poresizer 9320, Micrometrics Instrument Group, Norcross, GA) using crushed samples consisting of the broken cell walls and struts. The relative density, ρ_b/ρ_s, is the ratio of the bulk density (ρ_b) and the apparent density of struts (ρ_s) of RPCs. Compressive strength, σ_c, was measured by Instron 1195 universal testing machine using a crosshead speed 1.5 MPa/s. The cell size distributions were analyzed by Image-Pro Plus software (Media Cybernetics, Inc., Netherlands). The refractory was tested by standard cones correlation method (Shanghai Baoye Construction Corp., Ltd.).

The thermal shock resistance was tested by the water-quenching technique. The specimens were heated at a rate of 10 °C/min to the preset temperature and held for 10 min. Then the specimens were dropped into a water bath maintained at 20 °C. For comparison, the residual compressive strength of the specimens subjected to the thermal shock test was also measured under the same conditions and compared with the origin samples. In all measurements, six specimens were tested to obtain the average strength.

3. Results and discussion

In the previous paper,[11] we investigated the density and compressive strength for SiC RPCs recoated with Slurry 1, and revealed that the optimal sintering temperature of SiC RPCs is 1300 °C. In Fig. 1, the strut density and compressive strength of the SiC RPCs recoated with Slurry 2 are plotted as a function of heat treatment temperature of 1000–1300 °C in N_2. Apparently, the strut density of SiC RPCs increased as the sintering temperature increases. $MgO–A1_2O_3–SiO_2$ additives in the green body melt to form the liquid phase with low viscosity, which fill the pores, resulting into formation of more compact products. At the same time, the decomposition of PCS is taken place to form the SiC phase, which acts to bond between SiC particles in the strut layers. Both of these behaviors led the strut density to increase. As the strut density increases, the compressive strength of RPCs increases and reaches to the maximum value of 1.33 MPa at 1100 °C. When the sintering temperature was beyond 1100 °C, the compressive strength was found to decrease with increasing the temperature. Considering these results, we decided the sintering temperature for the SiC RPCs recoated with Slurry 2 to be 1100 °C. It is evident that this sintering temperature is lower by 200 °C than that for the SiC PRCs recoated with slurry 1. At the