



Fabrication of porous SiC/calcium hexaluminate composites



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ABSTRACT

Porous SiC/calcium hexaluminate (CA_6 , where C = CaO and A = Al_2O_3) composites were successfully prepared via two steps: (i) preparation of green body by direct foaming combined with pure calcium aluminate cement curing method at room temperature and (ii) sintering by in-situ formed CA_6 phase at relatively low temperature (1500 °C). The phase compositions, total porosity, compressive strength and the microstructure of porous SiC/ CA_6 composites with different composition and sintered at different temperatures were investigated. CA_6 phase with plate-like morphology was the main bonding phase in the porous composites, and a compressive strength of 14.0 ± 0.6 MPa was achieved with a total porosity of 82.9%.

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1. Introduction

Porous SiC ceramics have attracted more and more interests due to their low thermal expansion coefficient, high temperature stability, good oxidation resistance and excellent thermal shock resistance [1,2]. They have been considered as suitable candidate materials for catalyst supports [3,4], filters for hot gas or molten metal [5,6], high temperature membrane reactors [7], thermal insulating materials [8], and so on. However, the covalent nature of Si–C bond are so strong that the diffusion rate within SiC at an elevated temperature is extremely low, and it is difficult to manufacture self-bonded SiC ceramics below 2100 °C [2]. Several sintering additives, such as silica [9], cordierite [10], and mullite [11], were selected to reduce the sintering temperature [12]. Recently, CA_6 attracted more attention because of its excellent alkaline resistance, high stability in a reducing atmosphere, and low thermal conductivity [13]. It was used to produce high-strength and high-toughness ceramic composites through its plate-like grains which were developed in-situ at a relatively low temperature (<1600 °C) [14].

Direct foaming is a simple approach for the fabrication of porous materials. However, foam is a thermodynamically unstable system in the slurry, and the bubbles will become bigger or break up [15]. It is important to solidify the slurry in a short time. Several methods, such as gel-casting [16], starch consolidation [17], cement curing [18], sol-gel [19], etc. could solidify the slurry quickly. Calcium alu-

minate cement was widely used in refractory industry, and it could be hydrated with water and provide strength [20]. Furthermore, it can be used to react with $\alpha-Al_2O_3$ to form CA_6 .

In this study, SiC, $\alpha-Al_2O_3$ and pure calcium aluminate cement were used as raw materials to prepare porous SiC/ CA_6 composites. The green bodies were obtained by directing foaming method combined with cement curing process at room temperature. The total porosity was well controlled by changing the total mass of the powders. Effects of the total porosity and the mass percent of CA_6 on the compressive strength were studied. Effects of sintering temperature on phase composition and compressive strength of the porous SiC/ CA_6 composites were also studied. Microstructures of green body and sintered body were investigated by SEM.

2. Experimental procedure

SiC powders (98%) with an average particle size of 3.5 μm and alumina powders (99%) with an average particle size of 1.5 μm were used as main raw materials. Tetramethylammonium hydroxide (TMAH, analytically pure) was selected as aqueous dispersant, and triethanolamine dodecyl sulfate (chemically pure) was selected as foaming agent. Cement of Secar71 (Kerneos aluminate technologies Co., Ltd., China) was used as binder. The compositions of Secar71 are listed in Table 1.

SiC, Al_2O_3 , Secar71, TMAH, deionized water, and foaming agent, together with alumina balls, were poured into a ball milling tank. The sample design is listed in Table 2. After milling at the rate of 300r/min for 60 min, the tank was full of foaming slurry. Then the foaming slurry was diverted and casted into several plastic molds.

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Table 1
Compositions of Secar71.

Chemical composition	Content (mass%)
Al ₂ O ₃	69.9
CaO	29.6
SiO ₂	0.3
Fe ₂ O ₃	0.12

Several wet green bodies were obtained after 24 h. They were dried in an air oven at 110 °C for another 24 h. At last, the dried green bodies were heated in Ar with a heating rate of 5 °C/min and kept at 1300–1600 °C for 3 h.

The solid content of the slurry was controlled at 75 mass% for all the samples. The volume of the foaming slurry was controlled to be same by the volume of the tank. The sample compositions were designed and listed in Table 2. The masses of SiC, Al₂O₃ and Secar71 were calculated by the final composition of SiC and CA₆ based on the hypothesis that Al₂O₃ and Secar71 would transformed into CA₆ completely.

Open porosity of the dried green samples was determined by the Archimedes drainage method using kerosene as the medium. Bulk density of the sintered samples was determined by the Archimedes method using deionized water as the medium. Theoretical density of the sintered samples was determined in terms of the Chinese Standard GB/T 5071–2004. Total porosity of the sintered samples was calculated based on the following equations:

$$\pi_t = \frac{\rho_t - \rho_b}{\rho_t} \times 100\%$$

where ρ_t is theoretical density of the material, and ρ_b is the bulk density. Compressive strength was calculated from the maximum value of load divided by suffering area, and the sample size for testing was 40 mm × 40 mm × 40 mm. Microstructures of the samples were observed by scanning electron microscopy (SEM, PHILIPS-XL30). Phase identifications of the samples were determined by X-ray diffraction (XRD, D8 advance, Bruker, Germany).

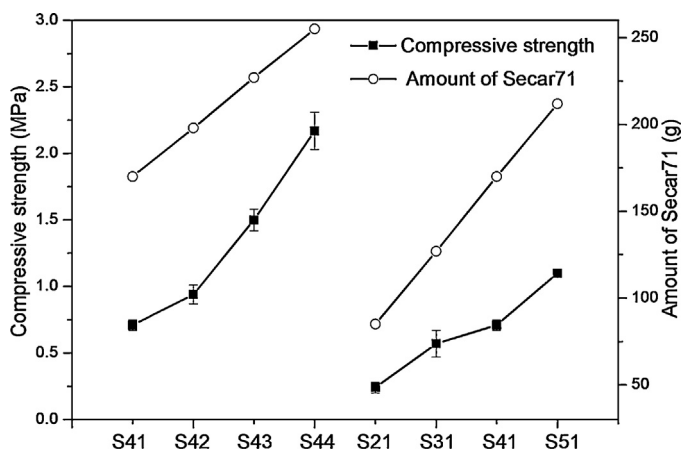
3. Results and discussion

3.1. Green body

Fig. 1 shows the compressive strength of the dried green bodies with different amount of Secar71. The open porosity of the dried green bodies is listed in Table 2. From S41 to S44, with the increasing of Secar71 amount and the decreasing of open porosity, the compressive strength increased. From S21 to S51, with the increasing of Secar71 amount and the relatively steady open porosity, the compressive strength also increased. But the slope of the later curve was smaller than the former one. Both amount of Secar71 and the porosity could affect the compressive strength of the green bodies, and the amount of Secar71 was the main reason. The compressive strength of the green body was provided by the hydrates which formed due to the reaction between Secar71 in the slurry and water.

Table 2
Sample design, the corresponding compositions, and open porosity of dried green bodies.

Sample No.	Starting materials (g)				Theoretical percent of CA ₆ (mass%)	Open porosity of dried green bodies (%)
	SiC	Al ₂ O ₃	Secar71	Total		
S41	900	430	170	1500	40	80.8
S42	1050	502	198	1750	40	76.1
S43	1200	573	227	2000	40	69.7
S44	1350	645	255	2250	40	65.9
S51	750	538	212	1500	50	80.6
S31	1050	323	127	1500	30	81.0
S21	1200	215	85	1500	20	81.3

**Fig. 1.** Compressive strength and Secar71 content of dried green bodies.

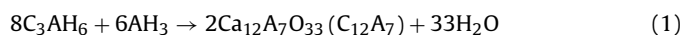
More hydrates would form with more cement. With the increasing quantity of Secar71 in the slurries with a constant volume, the compressive strength of the green bodies increased from 0.6 ± 0.1 MPa to 2.2 ± 0.1 MPa.

Fig. 2 shows the fracture morphology of green body (S41). As seen from Fig. 2(a), the pores were spherical, and most of the pores had diameters of smaller than 200 μm. One or two “windows” existed on the wall of each pore, and the “window” size was smaller than 30 μm. In Fig. 2(b) and (c), walls were packed by SiC and Al₂O₃ particles which were bonded by the hydrates of the cement.

3.2. Reaction bonding behavior at different sintering temperatures

Fig. 3 shows the XRD patterns of sample S41 treated at different temperatures for 3 h. SiC was the major phase for all of the five patterns. The green body after drying contained Al₂O₃ and some hydrates (3CaO·Al₂O₃·6H₂O (C₃AH₆) and Al(OH)₃ (AH₃)) besides SiC. When the green body was heat treated at 1300 °C or 1400 °C, a plenty of CaAl₂O₇ (CA₂) and CaAl₂O₄ (CA) formed and a small number of CA₆ could also be detected at 1400 °C. At 1500 °C, the amount of Al₂O₃ decreased abruptly and more extensive CA₆ peaks appeared. It can be inferred that reaction completely finished at 1500 °C by comparing with the phase compositions at 1600 °C.

With increasing temperature, a series of reactions occurred at the bonding areas. At temperatures not higher than 1300 °C, C₁₂A₇, CA and CA₂ had formed accomplishing with losing water, as shown in equations of (1)–(5) [20]. At 1400 °C, CA₆ began to form with the reaction between CA₂ and Al₂O₃. When the temperature increased to 1500 °C, more CA₂ and Al₂O₃ were consumed, and more CA₆ formed. Due to the difficulty of solid state reaction, there was a very small amount of residual Al₂O₃ at 1500 °C and 1600 °C.



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