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Preparation of biomorphic silicon carbide–mullite ceramics using molten salt synthesis

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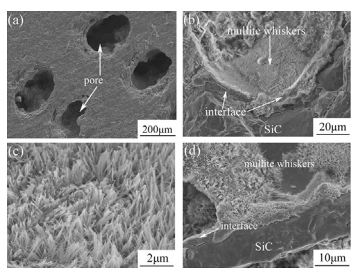
HIGHLIGHTS

- Biomorphic silicon carbide–mullite ceramics were prepared.
- An oxidation–dissolution mechanism was proposed to explain the coating formation.
- The unique structure has potential application in hot gases filter.

GRAPHICAL ABSTRACT



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ABSTRACT

Biomorphic silicon carbide–mullite ceramics were prepared from beech wood using liquid Si infiltration and molten salts synthesis. The resulting mullite whiskers coating, as well as the growth mechanism in molten $Al_2(SO_4)_3$–Na_2SO_4 environment, have been investigated using scanning electron microscopy (SEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR) techniques. The biomorphic SiC ceramics derived from the beech wood template have coarse pore walls consisting of β-SiC grains with diameters ranging from 5 μm to 20 μm. After the molten salts reactions between biomorphic SiC substrate and mixture molten salts ($Al_2(SO_4)_3$–Na_2SO_4), porous Silicon carbide–mullite ceramics with cilia-like microstructure were obtained. This unique structure has potential application in hot gases filters. An oxidation–dissolution cycle was proposed to explain the mullite whiskers growth in molten salts environment.

1. Introduction

Porous silicon carbide ceramics are some of the suitable materials for hot gases filter because of their desirable properties such as high temperature stability, low thermal expansion coefficient, high corrosion resistance, high thermal conductivity and excellent mechanical strength [1]. Furthermore, porous SiC ceramics are very low cost, which is benefit for the application in industry field. For example, wall-flow silicon carbide filter is the main material in diesel particulate filter (DPF) field, and the statistics of papers published in society of automotive engineers (SAE) display that more than 60% DPF selected porous SiC ceramics [2]. However, porous SiC ceramics fabricated by traditional procedure could not separate nano-particles from exhaust emission efficiently because the diameter of particulate is much less than the average pore diameter of SiC ceramics.

Nature is a great and successful laboratory ever existed. Through evolution, nature has experimented with various solutions to its

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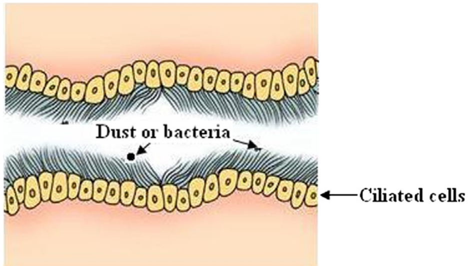

Fig. 1. The schematic diagram of bronchial surface.

challenges and has improved the successful ones, which provide ready answers to scientific and technical problems and inspire us with a series of novel designs and high-performance structures [3]. It is well known that human respiratory system has excellence filtration performance, the dust or bacteria could be filtered when the air flow through the bronchial surface, only the clean air enter lungs for breathing. The schematic diagram of bronchial surface is shown in Fig. 1. A large number of the ciliated cells grow on the surface of bronchial wall, and every ciliated cell has about 300 cilia with the lengths of six microns [4]. The harmful substance, such as dust, bacteria could be captured by these cilia. Inspired by this microstructure that is formed by natural evolution, we want to design cilia-like porous ceramics surface, and this novel structure could separate ultrafine particles efficiently.

Wood is a naturally grown composite material of complex hierarchical cellular structure, and composed of elongated tubular cells aligned with the axis of the tree trunk and growth ring structures [5,6]. The tubular cells of wood with a preferential orientation in axial direction offer the possibility to use liquid Si infiltration (LSI) techniques to transform the bioorganic wood structure into an inorganic ceramic material with tailored physical and mechanical properties [7,8]. The formation of biomorphic SiC ceramics included two step processes: (a) porous carbon template obtained by wood pyrolysis, (b) the conversion of carbon to ceramic using liquid Si infiltration technique. A number of studies have been reported on the preparation of wood-derived porous SiC ceramics and microstructure conversion [9,13]. As a new ceramic powder synthesis method, molten salt reaction (MSR) has been employed

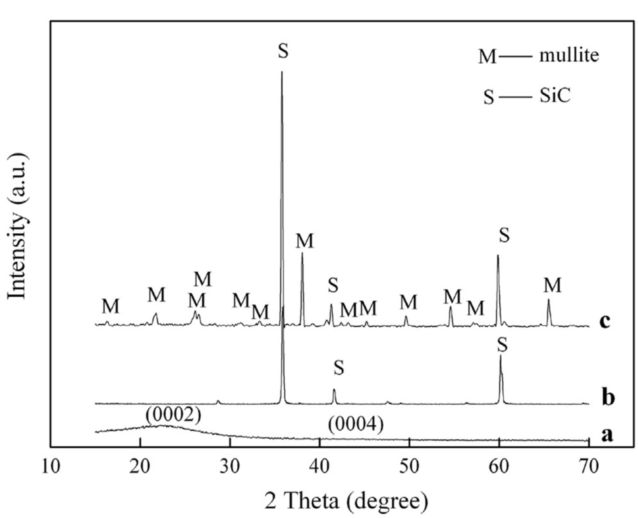

Fig. 2. XRD patterns of (a) charcoal, (b) biomorphic SiC, (c) biomorphic silicon carbide–mullite ceramics.

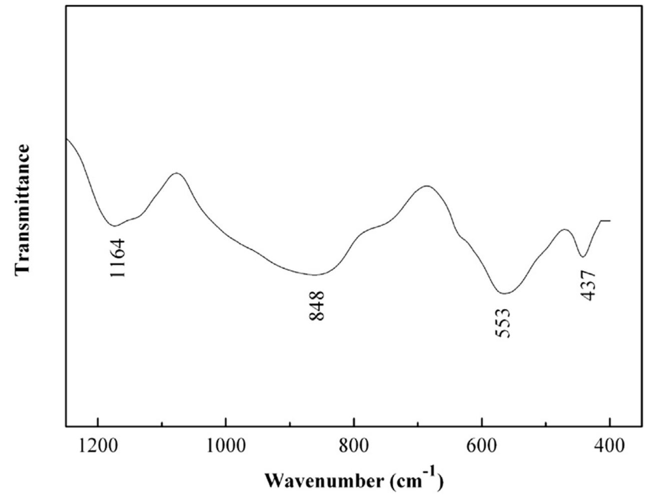

Fig. 3. Infrared spectra of biomorphic silicon carbide–mullite ceramics.

to synthesize ceramic powders because it decreases reaction temperature and gives powders of homogeneous morphology [14–16]. In our previous research, mixture molten salts ($Al_2(SO_4)_3 + Na_2SO_4$) could react with SiC ceramics to form mullite phase, and mullite crystal grow along one crystal plane firstly and developed into fibrous microstructure in molten salts system [17]. The objective of the present work is to prepare silicon carbide–mullite ceramics with cilia-like microstructure from beech wood by LSI and MSR processes. This unique microstructure has potential application in gases filter.

2. Experimental procedure

2.1. Material preparation

The wood pieces with 10 mm × 10 mm × 50 mm were cut from beech and dried at 105 °C for more than 24 h, which were divided to axial samples and radial samples according to the direction of length perpendicular and parallel to the direction of wood growth. The samples were pyrolyzed at 800 °C for 4 h with flowing N_2 gas protection to prepare the carbon templates, and then, packed in silicon powder and heated at 1550 °C for 1.5 h to form SiC in N_2 atmosphere. Finally, heated the furnace up to 1700 °C and used the mechanical vacuum pump to get rid of remnant silicon. Thus, the biomorphic SiC ceramics were obtained after the furnace was cooled down to room temperature.

The biomorphic SiC ceramics were treated in molten salts system, this process is described as follow: Firstly, $Al_2(SO_4)_3$, Na_2SO_4 and biomorphic SiC ceramics were weighed accurately according to a ratio of 9:10:1 in wt.%, and the mixed powders ($Al_2(SO_4)_3 + Na_2SO_4$) was grinded in a ceramic mortar for 30 min. Secondly, the biomorphic SiC samples was placed in the bottom of alumina boat and covered by the mixed powders ($Al_2(SO_4)_3 + Na_2SO_4$), and then slowly heated to 900 °C for 1 h and cooled down to room temperature in a maffle furnace. Subsequently, the samples were boiled in distilled water to remove the unreacted sulfate. Finally, the biomorphic silicon carbide–mullite ceramics were obtained after further drying. Aluminum sulfate ($Al_2(SO_4)_3$, AR, Xi'an Chemical Reagent Co.) and sodium sulfate (Na_2SO_4, AR, Xi'an Chemical Reagent Co.,) were used as raw materials in the present study.