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Effect of holding time on the basic properties of biomorphic SiC ceramic derived from beech wood

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Abstract

Liquid silicon infiltration processing to manufacture biomorphic silicon carbide is a low-cost route and the resulting products have high strength. Holding time is an important factor to fabricate biomorphic ceramics with various strengths and open porosity. In this study, biomorphic SiC ceramics were prepared by infiltrating liquid Si into biocarbon template from beech at 1550 °C for different reaction time (holding time I), and sequentially removing Si at 1700 °C for different time (holding time II). The resulting products have coarse surfaces of the pore walls because of recrystallization of formed SiC. The porosity decreases and the bending strength and fracture toughness enhances as prolonging reaction time. Both bending strength and fracture toughness generally decline, while open porosity increases and the density decreases with removing of unreacted Si in the samples. While removing Si time is prolonged from 20 min to 60 min, the average bending strengths of the axial and radial samples decline by 41.3% and 54.4%, respectively. The average fracture toughness decreases by 45.9% for the axial samples and by 49.7% for the radial samples. The key factors to affect properties of the resulting products are the formed SiC and the residual silicon. The difference of the porosity and density of the axial and radial samples is decided by the relationship of the direction of the channels of samples and the direction of infiltration.

Keywords: Biomorphic ceramics; Mechanical properties; SiC; Holding time

1. Introduction

Biomorphic ceramics/composites derived from wood, sawdust and papers have received more and more attention in the last decade [1–7]. Because wood is of renewable resource and using sawdust will minimize the environmental impacts, biomorphic ceramics which inherits microstructure of original wood and be of good prosperities were call as environment friendly material or ecoceramic material [8,9]. Biomorphic ceramics can be used for a variety of applications including filters and catalyst support, automotive components, tooling and wear components, armor, and lightweight, porous ceramics for aerospace systems [1]. In addition, biomorphic silicon carbide coated with bioactive glass or hydroxylapatite thin films have been proposed as an alternative to titanium and titanium alloy devices due to its low density, bio-inertness, interconnected porosity and improved mechanical properties [10,11].

Biomorphic ceramics or composites such as SiC, TiC, Al_2O_3, TiO_2 and Si–Mo–C, etc. have been fabricated by various methods [12–19]. The Biomorphic TiN/C ceramic were prepared by sol–gel impregnation and carbothermal nitridation [20].

Biomorphic SiC ceramics are first and extensively studied because of easy preparation processing and good properties. Previous methods converting pyrolysed biocarbon template into various SiC ceramic materials include the infiltration of liquid Si, gaseous Si, SiO and organosilicon compounds at high temperatures, sol–gel and carbothermal reduction processing and chemical vapor infiltration and reaction (CVI-R) processing [21–24]. However, gaseous silicon infiltration to manufacture biomorphic SiC needs long reaction time at high temperature [25]. Biomorphic SiC prepared by sol–gel impregnation and carbothermal reduction processing has low strength [26]. Compared with those processing, reactive infiltration with liquid Si of a porous biocarbon template obtained by pyrolysis of wood is a low-cost route to produce biomorphic SiC composites with open porosity: the synthesis rate is very high, and near-net shape production of complex forms. In addition, preliminary investigations have shown that the flexure strength of the biomorphic composites is comparable with that of commercial-grade reaction-bonded SiC

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[27–29] and the axial compressive strengths of the biomorphic SiC is higher than the strength of reaction bonded SiC (RBSC) and reaction formed SiC (RFSC) [30].

However, most of works on infiltrating liquid Si into biocarbon templates to manufacture biomorphic SiC or SiC/Si materials focused on fabrication and properties of the final products. As far as we know, there is little information on the relationship between the holding time and the basic properties of biomorphic SiC ceramics.

In fact, the biomorphic SiC should have various strength and open porosity for different application. For example, biomorphic ceramics/metal or ceramics/polymer composites can be manufactured via impregnated with liquid metals or polymer to porous biomorphic SiC. Biomorphic SiC for medical implant structures should need open pores to permit tissue in-growth. Except for choosing different kinds of original wood, holding time is another important factor to fabricate biomorphic ceramics with various strengths and open porosity. In the present study, biomorphic SiC ceramics were prepared by infiltrating liquid Si into the biocarbon templates derived from beech, we attempted to investigate the effects of holding time including reaction time and removing Si time on the mechanical properties of biomorphic SiC ceramics with open porosity.

2. Experimental

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, as shown in Fig. 1(a) and (b). The samples were impregnated with phenolic resin in order to reinforce the cell wall and to avoid distortion or cracking of samples during pyrolyzation process. The effects of phenolic resin on the basic properties of woodceramics and biomorphic SiC ceramics were discussed in another our paper [31].

Industrial silicon powder with a diameter range of 1–3 mm was used as a silicon source. All wood samples were pyrolyzed at 800 °C for 4 h with flowing N_2 protection to result in the biocarbon templates, and then, packed in silicon powder and heated at 1550 °C for 10, 30, 45, 60 and 90 min to form biomorphic SiC ceramics in a furnace. Finally, some samples were heated up to 1700 °C for 40 min to get rid of remnant silicon. Other samples with 60 min reaction time were removed remnant silicon for 20, 40 and 60 min. The weight ratio of the starting silicon powder to the biocarbon templates was 4:1. All the resulting samples were machined by a universal grinding machine.

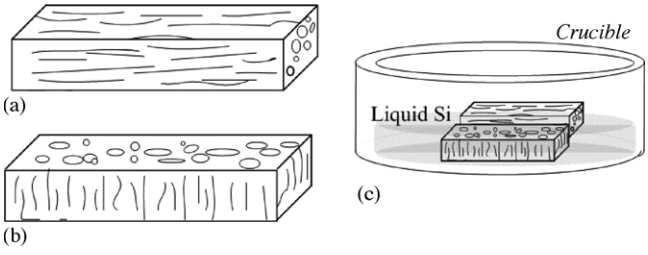

Fig. 1. Sketch of the axial sample (a), radial sample (b), and experimental disposal of samples (c).

2.2. Characterization

The XRD patterns of the biocarbon templates and the resulting products were recorded using X-ray diffractometer (D/MAX-RA, Rigaku, Japan) with nickel filtered Cu Kα radiation produced at 35 kV and 20 mA. The tested samples were broken and ground into powders in a carnelian mortar. The microstructure morphology was observed with scanning electron microscope (SEM) (S-2700, Hitachi, Japan) operated at 20 kV and 20 mA. Open porosity and density of resulting products were determined by the Archimedes method with distilled water as the liquid medium. Strength was tested by the three-point bending method with a 20 mm span. Fracture toughness was tested with the single edge notched beam (SENB) method. A notch with 2 mm depth and 0.28 mm width was cut in the middle of each SENB specimen. All the mechanical properties testing were carried out by a SANS-5104A machine (SANS Ltd., Tianshui, China) and crosshead speed was 0.5 mm/min.

3. Results and discussion

3.1. XRD analysis

Fig. 2 shows the XRD patterns of biocarbon templates and biomorphic SiC ceramics fabricated at various holding time I: reaction time. The axial samples were used in the XRD analysis. There are two main graphitic peaks corresponding to a broad (0 0 0 2) peak and a low intensity (0 0 0 4) peak (Fig. 2(a)), which suggests the biocarbon is amorphous. With increasing reaction time, the intensity of amorphous carbon decreased or even disappeared. The resulting products with 10 min reaction time consist

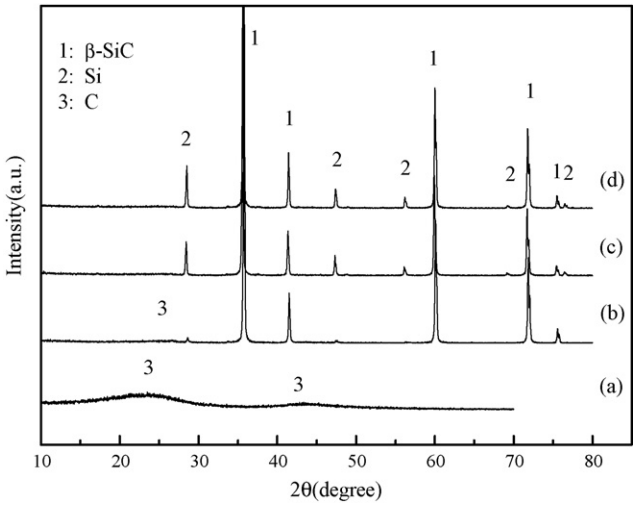

Fig. 2. XRD pattern of biocarbon template (a) and biomorphic SiC ceramics prepared for different reaction time: (b) 10 min, (c) 45 min, and (d) 90 min. The axial samples removed Si for 40 min were used in the XRD analysis.