Materials and Design 34 (2012) 528–532

Contents lists available at ScienceDirect

Materials and Design

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

Short Communication

Laminated biomorphous SiC/Si porous ceramics made from wood veneer

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ARTICLE INFO

Article history:
Received 24 January 2011
Accepted 4 May 2011
Available online 9 May 2011

ABSTRACT

Biomorphous SiC/Si porous ceramics with laminated structure are prepared from beech veneer and phenolic resin. The preparation involves carbonization under vacuum and reaction with melted silicon to obtain the biomorphous carbide template. X-ray diffraction confirms that the biomorphous SiC/Si porous ceramics are mainly composed of β-SiC, free silicon and residual carbon. Scanning election microscopy observations indicate a laminated structure and 1–10 μm microporous structures, which suggest retention of the native characteristics of the wood. This paper examines mechanical properties of the final composite in relation to the lamination, porous structure, and free silicon content. The bending strength of the ceramics decreases as the apparent porosity increases. The fracture toughness increases initially with apparent density and then decreases. The fracture toughness load–displacement curve presents a step-like pattern, which suggests that the laminated SiC/Si porous ceramics have high fracture toughness.

1. Introduction

Biomorphous SiC/Si porous ceramics synthesis from biomass materials has become an area of increasing interest mainly due to their natural microstructural features. A cellular SiC ceramic prepared from corn stems through a slow infiltration technique [1], and a beech biomorphous Si/SiC ceramics obtained with liquid Si infiltration [2]. Fey et al. [3] had discussed the stress distribution in biomorphous SiC ceramics, and a biomorphic porous SiC which prepared from carbonized millet had been studied by Wang et al. [4]. The specific microstructures of this material impart important properties such as high strength, stiffness [5], toughness, porous structures and anisotropic permeability [6] at low density.

Two carbide templates are used to prepare biomorphic SiC/Si porous ceramics at present. One is bulk wood carbonized at high temperature [7], and the other is sintered composite of medium-density fiberboard (MDF) and wood powder (or other biomass materials powder)/thermosetting resin [8]. However, the template made from bulk wood easily deforms and breaks because of anisotropy of the wood, which leads to the smaller size of SiC/Si porous ceramics. The MDF loses nearly all of the biomorphous properties of wood due to its small particle size.

Laminated ceramic has many superior characteristics, which might be of great value for impact-resistant material [9], high-temperature resistant exhaust gas filters, advanced microreactor, lightweight aeronautical materials and tooling and wear components, etc. [10]. But preparation of laminated biomorphic SiC/Si porous ceramics from native wood veneer has never been done. In this paper, a new type of laminated SiC/Si porous ceramic with biomorphous performance of wood is prepared and characterized on the basis of exploratory experiments [11]. This paper also examines the synthesis, microstructure characterization, phase evolution and mechanical properties of the ceramic.

2. Experimental procedure

2.1. Specimen preparation

Sliced beech veneer (150 mm × 150 mm × 0.5 mm) is used as the biomass material and phenolic resin (PF) with 52% solid content is the adhesive. The beech veneer is dried to 8% moisture content at 60 °C and the glue spread is 160 g m^{-2}. More than 50 tiers of beech veneer stacked with parallel texture are hot-pressed into beech veneer/PF resin plywood under 5 MPa at 160 °C for 10 min (as shown Fig. 1). The resulting plywood is carbonized in a high-temperature vacuum sintering furnace at a heating rate of 2 °C min^{-1} until 350 °C, then at 5 °C min^{-1} until 1200 °C, and finally hold for 2 h at 1200 °C. Afterward, it is cooled to ambient temperature in the furnace. Thus, the laminated carbide template is obtained.

The carbide templates are cut into 60 mm × 60 mm squares, picked in silicon powder, heated together at 1420 °C in a vacuum furnace at 5 °C min^{-1} heating rate and held for 30 min, and then they are cooled to ambient temperature at 5 °C min^{-1} rate. Subsequently, some samples are retreated at 1700 °C for 20–100 min to remove excess free silicon in the cells and cracks. The laminated SiC/Si porous ceramics with biomorphous are obtained. The process is shown in Fig. 2.

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doi:10.1016/j.matdes.2011.05.004

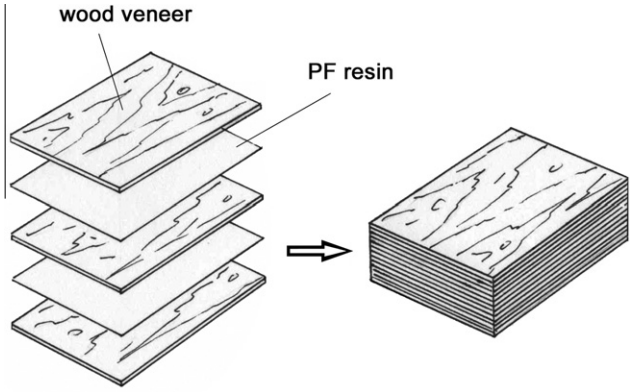

Fig. 1. Process of laminated wood veneer/PF resin composite.

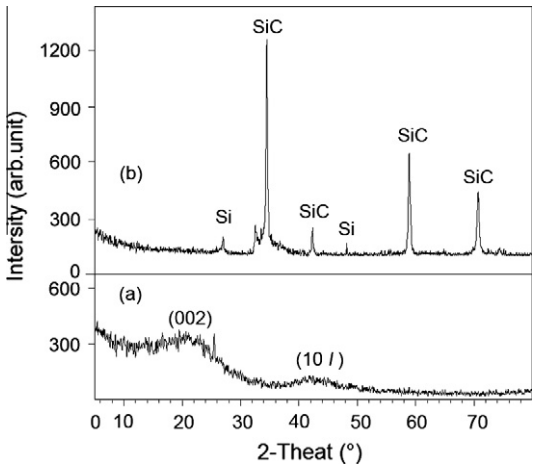

Fig. 3. XRD patterns of wood template and SiC/Si porous ceramics.

2.2. Characterization methods

The samples are cut into bars so that the long edge is parallel to fiber direction. An X-ray diffractometer (XRD; XD-2, China) is employed to investigate the degree of carbonization of the wood template and the phase of the SiC ceramics, scanning at the speed of $2^\circ\ min^{-1}$. The morphology of the wood carbide template and the final laminated SiC/Si porous ceramics are recorded and analyzed by a scanning electron microscope (SEM; JSM-35C, Japan) operated at 20 kV and 20 mA. Strength at room temperature is tested with a testing machine (Instron 8802, USA) by four-point bending (20/40 mm) according to ASTM C1161–90 [12], using $5 \times 6 \times 60\ mm^3$ specimens, the loading rate is $0.5\ mm\ min^{-1}$. The fracture toughness is tested with single edge-notched beam (SENB) method. The apparent density and the apparent porosity are measured by a density tester (MDMDY-300, China) and by Archimedes measurements.

3. Results and discussion

3.1. Phase evolution

The crystallinity of the composite changes during carbonization and infiltration [13]. Fig. 3 shows the XRD patterns of the carbide template and the laminated SiC/Si porous ceramics. Beech veneer is converted into amorphous carbon, and the PF resin becomes glassy carbon at high carbonization temperature [14]. No sharp peak appears in XRD pattern (Fig. 3a). The distinctive 0 0 2 peak is broad, and the 10*l* peak is almost invisible because it is difficult for the two kinds of carbon to completely graphitize [15]. The patterns of the infiltrated ceramics template are very different from this peak (Fig. 3b). Distinct peaks of β-SiC and Si in the pattern suggest that these substances comprise the main crystal phase. During infiltration, Si melts at 1410 °C and permeates through the longitudinal vessels and cell walls to form β-SiC, as described by Eqs. (1) and (2) [16].

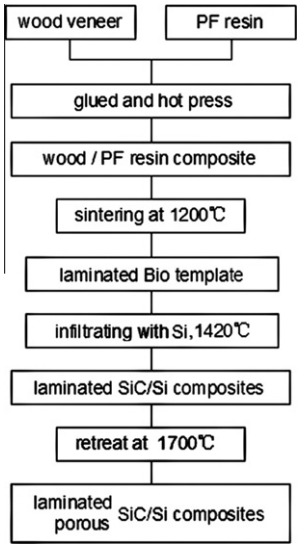

Fig. 2. Process of laminated SiC/Si porous ceramics from beech carbide template and melted silicon.

$$C_{amophous} + Si_{(l)} \rightarrow \beta\text{-SiC} \quad (1)$$

$$C_{glassy} + Si_{(l)} \rightarrow \beta\text{-SiC} \quad (2)$$

The final laminated SiC/Si ceramic is a composite. The free silicon cannot be excluded thoroughly, since it is present in the pores and cracks even if the template has been retreated at high temperature (1700 °C). But the diffraction peak near at $2\theta = 35.64^\circ$ for the α-SiC has not almost detected in the XRD pattern because the sintering temperature is lower than 2000 °C, which does not lead to the transformation of β-SiC to α-SiC. However, residual carbon still remains in the sample. This suggests that the laminated ceramic is a three-phase composite material consisting of β-SiC, free silicon and carbon. These observations are consistent with a previous study on biomorphous SiC/Si porous ceramics prepared from wood powder [17]. But the contents of free silicon and residual carbon will be reduced with a longer retreat time, because they not only continue to react each other to form SiC but also are gasified at high temperature. For this reason, the residual carbon almost disappears at last as well as very few of free silicon still remain in the pores and cracks. The main phase of the composite is β-SiC.

3.2. Microstructure characterization

An important objective of this work is to produce a SiC/Si ceramic that has a laminated structure and the biological properties of wood. The density, hardness and pore structure of amorphous carbon of the carbide template are different from those of glassy carbon resulting from the laminated structure [11]. In the same token, the layered structure of the interface can be formed in the SiC/Si composite when melted silicon reacts with amorphous carbon [18].