Available online at www.sciencedirect.com

ScienceDirect

Ceramics International 40 (2014) 1939–1944

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Mechanical properties of SiC/SiC composites fabricated by PIP process with a new precursor polymer

Zheng Luo*, Xingui Zhou, Jinshan Yu, Ke Sun, Fei Wang

Science and Technology on Advanced Ceramic Fibers and Composites Laboratory, National University of Defense Technology, Changsha 410073, China

Received 20 May 2013; received in revised form 18 July 2013; accepted 22 July 2013
Available online 27 July 2013

Abstract

Three dimensional four-directional (3D4d) silicon carbide (SiC) fiber reinforced SiC matrix composites (SiC/SiC) were fabricated by the precursor impregnation and pyrolysis (PIP) process with a new precursor polymer, liquid polycarbosilane with active Si–H and 3CHåCH$_2$ groups (LPVCS). The densification, mechanical properties and microstructure of the SiC/SiC composites were studied. The composite processed with LPVCS exhibited excellent physical and mechanical properties. The density and the porosity of the composites were 2.16 g/cm^3 and 6.7%, respectively. The flexural strength of the composites was 619.4 MPa and fracture toughness was 29.1 MPa m$^{1/2}$. The results indicated that LPVCS was a kind of potential precursor polymer for the preparation of SiC/SiC composites.

Keywords: C. Mechanical properties; New precursor polymers; PIP; SiC/SiC composites

1. Introduction

SiC/SiC composites are excellent thermal structural materials owing to their outstanding high-temperature strength, fracture toughness, chemical inertness, and low density [1–4]. Currently, the SiC/SiC composites are the leading candidates for aircraft engine hot-section components [5–8]. The precursor impregnation and pyrolysis (PIP) process is one of the main preparation processes of the SiC/SiC composites and has been widely employed because of its advantages in microstructure control, low costs and large-scale components fabrication with complicated shapes [9–10].

Polycarbosilane (PCS) is a conventional precursor polymer for SiC matrix, and it could be solved in Xylene for infiltrating into SiC fiber performs. Pores and cracks are formed due to gas evolution and volumetric shrinkage of the precursor polymer during pyrolysis which leads to the performance degradation of the composites [11–14]. Many efforts have been done to search the new precursor polymers to improve the mechanical properties of the SiC/SiC composites due to the shortcomings of poor efficiency of impregnation, low ceramic yield and small pores existing in the matrix. Kotani et al. [15–16] prepared SiC/SiC composites by the PIP process with the matrix precursor, liquid polycarbosilane with a lot of functional Si–H bonds (PVS). The PVS was applied because of its advantages of sufficient stability at ambient temperature, low viscosity, and continuous thermosetting behavior. It showed that the density and process efficiency of the SiC/SiC composites were increased and the flexure strength was 602 MPa. Kohyama et al. [17] developed a near-stoichiometric SiC matrix by mixture of PCS and polymethylsilane (PMS). It demonstrated remarkable improvements in tensile properties and fatigue limits (at 1300 °C) of the SiC/SiC composites were attained. Nannetti et al. [18] prepared SiC/SiC composites with the matrix precursor, allylhydridopolycarbosilane (AHPCS), contributing to great increase of composites' thermal diffusivity under high-temperature pyrolysis treatment at 1700 °C, which promoted polymer derived SiC matrix crystallization.

A new precursor polymer called LPVCS which is prepared by taking 2,4,6,8-tetravinyl-2,4,6,8-tetramethylcyclotetrasiloxane (V4) as curing agent and liquid polycarbosilane as starting materials is used for the preparation of high performance SiC/SiC composites.

*Corresponding author. Tel.: +86 731 84576397; fax: +86 731 84573165.
E-mail address: lzzw2003@sina.com (Z. Luo).

0272-8842/$ - see front matter
http://dx.doi.org/10.1016/j.ceramint.2013.07.102

LPVCS exhibits a low viscosity and good stability at room temperature, as well as a high ceramic yield and low cost. 3D4d braiding performs are composed of four directional yarns which are braided with the same braid angles in the interior of the material (Fig. 1). The 3D4d braided performs as reinforcements had the advantages of through-thickness reinforcement, low delamination tendency, high damage tolerance and near-net-shape manufacturing [19]. In this work, the 3D4d SiC/SiC composites were fabricated with two different precursor polymers, LPVCS and PCS, for comparison. The density, mechanical properties and microstructure of the SiC/SiC composites were studied.

2. Materials and experimental procedure

LPVCS and PCS were provided by National University of Defense Technology, China. Polymer-derived KD-I SiC fiber bundles provided by National University of Defense Technology were used as reinforcements. The PyC coating was prepared on the SiC fiber fabric with a thickness of about 500 nm (Fig. 2) by chemical vapor deposition method at 900 °C in an acetylene atmosphere. The fiber volume fraction of the 3D4d SiC fiber performs was about 46.5%. The SiC fiber performs were impregnated with LPVCS or PCS solution by a vacuum infiltration method and pyrolyzed at 1100 °C in an inert Argon atmosphere. The impregnation and pyrolysis process were repeated until weight increase was less than 1%. Four sets of specimens with different precursors and interfaces were prepared (Table 1).

The densities and open porosities of the SiC_f/SiC composites were measured by Archimede's method. The flexural strength was measured by three-point-bending test on a GTM91100 apparatus. The dimension of the samples for the test was 4.0 mm $(B)\times$ 3.0 mm $(H)\times$ 60.0 mm (L). The span length was 30 mm, and the crosshead speed was 0.5 mm/min. The fracture toughness of the SiC/SiC composites was determined with the single edged-notched beam method (SENB). Cross section morphology was observed by X-ray tomographic techniques, samples were scanned using a Metris X-tek 320 kV source at the Henry Moseley X-ray Imaging Facility in the University of Manchester. The microstructure of the fracture surfaces was characterized by a JSM-5600LV scanning electron microscope (SEM) and a JE-200 transmission electron microscopy (TEM).

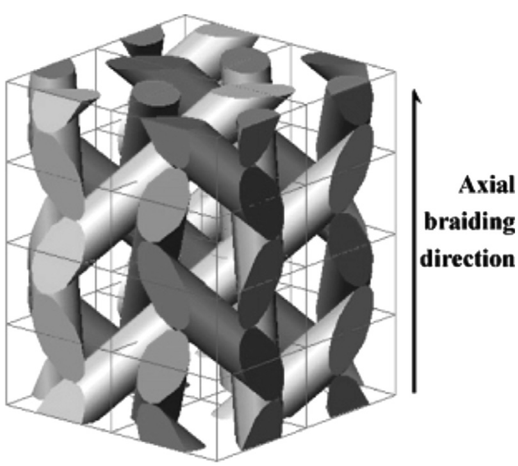

Fig. 1. Schematic of 3D4d preform.

Table 1
List of specimens with different precursors and interfaces.

Specimen	Interface coating	Precursor	Preparation cycle
1#	–	LPVCS	10
2#	–	PCS	14
3#	PyC	LPVCS	10
4#	PyC	PCS	14

Table 2
Basic characteristics of LPVCS and PCS.

	LPVCS	PCS
State at RT	Liquid	Solid
Density (g/cm^3)		
As-received	1.04	1.15
After pyrolysis	2.42	2.34
Impregnation	Without solvent	Solved in Xylene
Viscosity (mPa s)	20	25
Volume change	Shrinkage	Expansion
Ceramic yield (%)	59.5	31.5

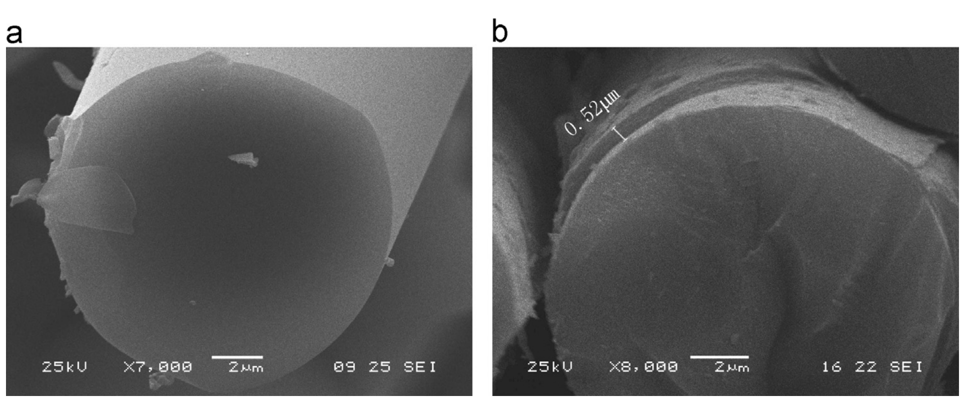

Fig. 2. Microstructure of the SiC fibers: (a) before the CVD method; (b) after the CVD method.