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Effect of pyrolysis temperatures on the performance of SiC_f/SiC composites

Yuxin Chai [a,b,*], Huayu Zhang [a], Xingui Zhou [b], Bei Yang [b]

[a] Shenzhen Key Laboratory for Advanced Materials and Department of Material Science and Engineering, Shenzhen Graduate School, Harbin Institute of Technology, University Town, Shenzhen, Guangdong, 518055, China
[b] Science and Technology on Advanced Ceramic Fibers and Composites Laboratory, National University of Defense Technology, Changsha, Hunan, 410073, China

HIGHLIGHTS

- Three-dimensional SiC_f/SiC composites were fabricated by the polymer infiltration and pyrolysis (PIP) method at different pyrolysis temperatures (1100, 1300 and 1500 °C).
- The effect of the pyrolysis temperature on the thermal and mechanical properties of the SiC_f/SiC composites was investigated by a laser flash method, three-point bending test and single-edged notch beam method.
- The interface shear strength of SiC_f/SiC composites was tested through a single-fiber push-out test system.

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ABSTRACT

Continuous silicon carbide fiber reinforced silicon carbide matrix composites (SiC_f/SiC) are promising candidate materials for nuclear applications. In this paper, three–dimensional (3D) SiC_f/SiC composites were fabricated using the polymer infiltration and pyrolysis (PIP) process at different pyrolysis temperatures (i.e. 1100, 1300 and 1500 °C). The effect of the pyrolysis temperature on the thermal and mechanical properties of the SiC_f/SiC composites was investigated with a laser flash method, a three–point bending test and a single–edged notch beam method. The results indicated that the thermal diffusivity of the SiC_f/SiC composites improved considerably with increasing pyrolysis temperatures, due to a higher degree of crystallization in the matrix. Additionally, as the testing temperature increased, the thermal diffusivity of the SiC_f/SiC composites gradually decreased. With increasing pyrolysis temperatures, the mechanical properties of the SiC_f/SiC composites first increased and later decreased. The SiC_f/SiC composites fabricated at 1300 °C had the highest average flexural strength and fracture toughness, i.e. 535.6 MPa and 17.3 MPa $m^{1/2}$, respectively.

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1. Introduction

SiC_f/SiC composites are promising candidate materials for high–temperature structural applications, such as gas turbines and aerospace propulsion systems [1–3], as well as nuclear applications, such as fusion and advanced fission reactors [4,5], due to their good high temperature resistance, corrosion resistance, thermal–shock resistance, oxidation resistance, relatively low induced radioactivity and high dimensional stability in addition to their performance stability after exposure to high energy radiation [6–9]. In recent years, many researches have been conducted on SiC_f/SiC composites. There are several methods for fabricating SiC_f/SiC composites, such as polymer infiltration and pyrolysis (PIP) [10], chemical vapor infiltration (CVI) [11], nano–infiltrated transient eutectoid (NITE) [12] and reaction infiltration (RI) [13], etc. SiC_f/SiC composites fabricated via the traditional PIP process have many defects, such as high porosity and a low degree of matrix crystallization, because the poor heat–resistance of the first generation SiC fibers limits the temperatures available for preparing SiC_f/SiC composites [14,15].

The three generations of SiC fiber differ in their composition and heat resistance. The Nicalon NL–200, Hi–Nicalon and Hi–Nicalon–S of Nippon Carbon Co., Ltd. belong to the three generations of SiC fiber, and their maximum temperature limits are 1200, 1600 and

* Corresponding author at: Shenzhen Key Laboratory for Advanced Materials and Department of Material Science and Engineering, Shenzhen Graduate School, Harbin Institute of Technology, University Town, Shenzhen, Guangdong, 518055, China.
E-mail address: kevinoldstar@qq.com (Y. Chai).

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Table 1
General properties of the as–received KD–II SiC fibers.

Properties	C/Si mole ratio	Oxygen (wt%)	Density (g cm^{-3})	Diameter (μm)	Strength (GPa)	Modulus (GPa)
KD–II	1.41	1.3	2.7	12–14	3.0	278

1800 °C, respectively [16–18]. However, the researches have barely mentioned the development of SiC fiber in China. Only the National University of Defense and Technology has developed the KD–I and KD–II SiC fibers, which belong to the first and the second generation SiC fibers, respectively. A certain amount of oxygen is introduced into KD–I SiC fiber during the stabilization process, which forms a SiC_xO_y phase in the fiber. In order to inhibit the decomposition of SiC_xO_y, which leads to a sharp decrease to the fiber's performance, KD–I SiC fiber could not be used at temperatures exceeding 1100 °C [19]. Therefore, the KD–I SiC fiber has poor heat resistance, which limits the fabrication temperature of its reinforced SiC_f/SiC composites. As processing conditions improved, the KD–II SiC fiber is irradiated with an electron beam to prevent extra oxygen from being introduced. Thus, the fabrication temperature of KD–II SiC fiber (approximately 1350 °C) is higher than that of KD–I SiC fiber. This property enhances the heat–resistance of KD–II SiC fibers. Therefore, the degree of crystallinity and the density of the matrix could be increased by increasing the pyrolysis temperature, which effectively fixes the defects in the PIP process [20,21].

In this paper, a mixture of 2,4,6,8–tetravinyl–2,4,6,8–tetramethyl cyclotetrasiloxane (V4) and liquid polycarbosilane (LPCS), known as LPVCS as the precursor [22,23], was used as the ceramic liquid precursor of the SiC matrix, KD–II SiC fibers were used as the reinforcement, and a PyC coating was used as the interfacial layer. Different SiC_f/SiC composites were prepared by increasing the pyrolysis temperature (i.e. 1100 °C, 1300 °C and 1500 °C) during the PIP process. The effect of the pyrolysis temperature on the mechanical and thermal properties of the SiC_f/SiC composites was investigated. ‘

2. Materials and methods

Three–dimensional and four–directional KD–II SiC braided fibers were used as reinforcement [24,25]. The KD–II SiC fibers used in this study were produced using Yajima's procedure [26]. The general properties of KD–II SiC fibers are shown in Table 1 [19]. The 3D and four–directional KD–II SiC braided fiber knitted by four–step method involved the knitting of the yarns into a matrix by rows and columns. Each yarn was independently controlled by a carrier which alternatively moved back and forth along each row and each column. The yarns were lined in four directions. The main parameters of the prefabricated piece are listed in Table 2.

Table 2
Parameters of 3D four–directional prefabricated piece.

Size (mm)	550 (length) × 60 (width) × 4.5 (thickness)
Structure	three–dimensional and four–directional
Yarn specification	1K × 1 strand
Circumferential stitch (mm/10 stitch)	15
Longitudinal stitch (mm/10 stitch)	35–45
Total weight (kg)	0.471
Volume fraction (%)	47.5

LPVCS (viscosity of 20 mPa·s at 25 °C, provided by NUDT) was used as a ceramic precursor and is a 0.6:1 weight ratio mixture of V4 and LPCS [22,27]. A single PyC coating (200–500 nm) was used as the SiC_f/SiC composite interfacial layer. SiC_f/SiC composites were fabricated by the PIP process. The process flow is shown in Fig. 1.

The bulk density and open porosity of the SiC_f/SiC composites were determined by Archimedes' principle (ASTM D792). The flexural strength and fracture toughness of the SiC_f/SiC composites were characterized through the three–point bending test (ASTM C1341-06) and the single–edged notch beam method (SENB) (ASTM E1820-11E2), which were conducted with a WDW–100 electronic universal testing machine. The three–point bending test samples were 55 × 4 × 3 mm^3 with a span of 50 mm and a cross–head speed of 0.5 mm min^{-1}. The single-edged notch samples were 35 × 6 × 3 mm^3 with a span of 30 mm, a notch depth of 3 mm and a cross–head speed of 0.05 mm·min^{-1}. Five specimens were tested to estimate the scatter in the mechanical tests. The interfacial shear strength was tested through a single–fiber push–out test system, which was fabricated by the researchers of Harbin Institute of Technology from an HIT–100 micro debonding tester. The schematic diagram is shown in Fig. 2. The single–fiber push–out test samples were 2 × 2 mm^2 with a mechanical shearing thickness of 100–150 μm [14].

The morphology of the fracture surface was analyzed by scanning electron microscopy (SEM) using a JMS–5600 field emission scanning electron microscope (JEOL Ltd.). The crystal structures of the as–received SiC fiber and SiC matrix (which were ground into

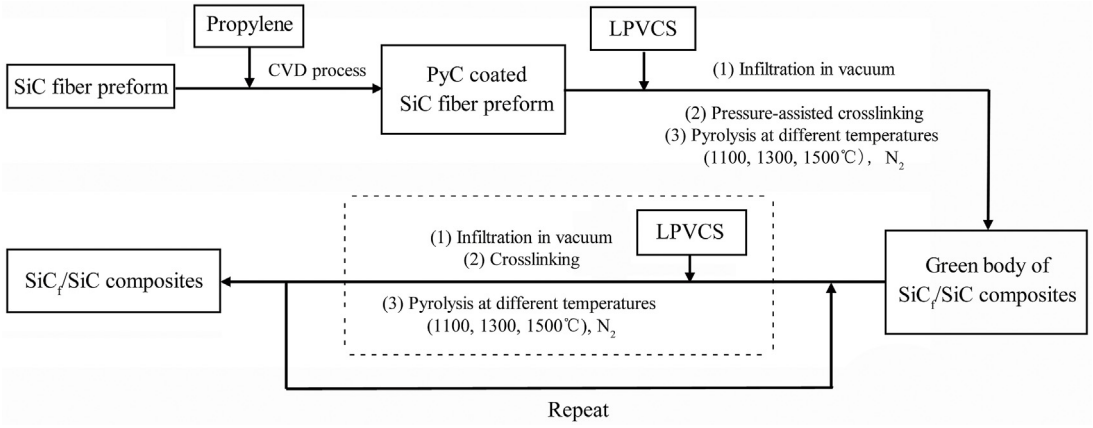

Fig. 1. Flow chart of the fabrication process of the SiC_f/SiC composites.

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