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Mechanical and microwave dielectric properties of KD-I SiC_f/SiC composites fabricated through precursor infiltration and pyrolysis

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Abstract

SiC fiber-reinforced SiC matrix (SiC_f/SiC) composites, which employ a new type of KD-I SiC fibers (provided by the National University of Defense Technology, China) with in situ pyrocarbon (PyC) coating on the surface of the fibers as reinforcements, are fabricated through precursor infiltration and pyrolysis (PIP). The characteristics of the fiber surface are evaluated by scanning electron microscopy, X-ray photoelectron spectroscopy, Auger electron spectroscopy, and transmission electron microscopy. The mechanical and dielectric properties of KD-I SiC_f/SiC composites are reported, and the effects of in situ PyC coating on the material properties are investigated. Results show that the KD-I SiC fiber has a special "skin (PyC phase)-core (Si–C–O phase)" structure. The composites possess excellent mechanical properties because of the in situ PyC coating on the surface of the fibers. The flexural strength and toughness are 268.8 MPa and 12.9 MPa $m^{1/2}$, respectively. However, the dielectric constants are also remarkable, which is disadvantageous to microwave absorbing applications.

Keywords: C. Mechanical properties; C. Dielectric properties; SiC_f/SiC composites; Precursor infiltration and pyrolysis

1. Introduction

The demand for microwave absorbing materials, which are functional materials widely used in civil and military domains, have been increasing continuously at present because of the progressive uses of microwaves. These materials should ideally feature low density, favorable mechanical properties, and appropriate dielectric properties for strong microwave absorption over a wide frequency range. Moreover, these materials should serve at high temperature environments, particularly for applications in stealth technology for aircrafts such as supersonic cruise missiles and the battle plan of high Mach numbers [1–4].

SiC fiber-reinforced SiC matrix (SiC_f/SiC) composites possess superior properties, such as excellent high temperature mechanical properties, good fracture and corrosion resistance, and thermodynamic stability [5,6]. Given these excellent characteristics, SiC_f/SiC composites are regarded as one of the most promising materials for structural applications at elevated temperatures. The excellent semiconductivity and relatively stable dielectric properties at elevated temperatures of these materials are also demonstrated in numerous practical and potential applications of high-temperature microwave absorption [7–9].

SiC fiber usually requires an appropriate coating on its surface to achieve promising reinforcement for composites because the interphase formed from the coating allows crack deflection, fiber pullout, and fiber/matrix (F/M) debonding, all of which provide excellent mechanical properties to SiC_f/SiC composites [10,11]. Different kinds of coatings have been suggested recently, such as carbon (C), boron nitride (BN), oxide and multilayer coatings ($(X-Y)_n$) [12–16]; however, carbon has been recognized as one of the most commonly used and effective coating materials. Aside from the favorable compatibility at the fiber/matrix interface, carbon layer

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provides an adjustable electrical resistivity to SiC fibers, which remarkably affects the dielectric properties of SiC_f/SiC composites [17].

The fabrication process is also one of the key factors that determine material properties. Concerning the manufacturing processes for the densification of fiber preforms, the most developed ones are chemical vapor infiltration (CVI) [18] and polymer infiltration and pyrolysis (PIP) [19,20]. Both techniques are based on the common principle of filling the porosity inside the fiber preforms with a SiC matrix resulting from decomposition of gaseous precursors (CVI) or pre-ceramic polymer precursors (PIP). General properties and issues of SiC_f/SiC composites currently in vogue are summarized in Table 1. The mechanical behavior is highly dependent on composite microstructure, fiber types, interphases and manufacturing methods.

In the present study, a new type of SiC fibers, namely, KD-I SiC fibers with pyrocarbon (PyC) coating on its surface, is employed as reinforcement for SiC_f/SiC composites. The PyC layer is obtained in situ during the SiC fiber fabrication process. The mechanical and dielectric properties of KD-I SiC_f/SiC composites fabricated through PIP are reported. The effects of the in situ PyC layer on material properties are also investigated.

2. Experimental procedure

2.1. Preparing KD-I SiC_f/SiC composites

KD-I SiC fibers, which were provided by the National University of Defense Technology, were produced following the route presented by Yajima [31], which involves three steps: (1) spinning the polycarbosilane (PCS) precursor in the molten state, (2) curing treatment to make the polymer fibers infusible, (3) and pyrolysis of the cured filaments in an inert atmosphere under different pyrolysis modes. KD-I SiC fibers were fabricated through the one-step pyrolysis mode. The length of the temperature constant area in the pyrolysis furnace was 600 mm. The rolling speed was 60 mm/s. Nitrogen gas flew in from both sides of the corundum tube. The N_2 flux was 2 L/min. The pyrolysis temperature was 1200 °C. The detailed preparation procedure could be found in Refs. [32–34]. The fibers had a mean diameter of 15 μm with 600 filaments in a bundle. The basic chemical composition was made of 34.7 at% silicon, 41.8 at% carbon, and 23.5 at% oxygen [35]. The typical characteristics of KD-I SiC fiber and other commercial SiC fibers are listed in Table 2.

The reinforcements used to prepare 2D SiC_f/SiC composites were plain-weave KD-I SiC fiber cloths. PCS, the precursor of the SiC matrix with a relative molecular mass of ~1300 and a softening point of ~210 °C, was synthesized in our laboratory. The 2D KD-I SiC_f/SiC composites were fabricated through PIP. The detailed process could be found in Ref. [10].

2.2. Analytical methods

Scanning electron microscopy (SEM) investigation was performed on a Hitachi FEG S4800 SEM (Hitachi Ltd., Japan) to analyze the morphology of SiC fibers and to observe the fracture surface of the composites. The specimens for transmission electron microscopy (TEM) observation were prepared by following the preparation procedure described by Appiah et al. [38]. JEOL JEM-2010 (JEOL Ltd., Japan) and Philips CM 200 FEG (Philips, the Netherlands) equipped with a Gatan imaging filter (GIF) system were used to characterize the cross section of the SiC fibers and the interfacial microstructure between fibers/matrices. The surface compositions of the SiC fibers were investigated by X-ray photoelectron spectroscopy (XPS) analysis using a VG ESCALAB MK II, with Al Kα radiation and calibrated against Au $4f_{7/2}$ and Cu $2p_{3/2}$ lines. The atom concentrations of carbon, silicon, and oxygen along the radial direction were measured by Auger electron

Table 1
Applications of carbon interphase in 2D-SiC_f/SiC composites.

Fiber type	Preparation process	Thickness of interphase (μm)	Mechanical properties (MPa)	References
Nicalon	CVI	0.1	145 (TS)	[21]
	CVI	0.13	420 (FS)	[22]
	PIP	0.04	350 (FS)	[23]
	PIP	0.04	136 (TS)	[24]
Hi-Nicalon	CVI	0.15	268 (TS)	[25]
	PIP	0.04	230 (FS)	[23]
	PIP	0.06	210 (TS)	[26]
			190 (FS)	
Tyranno SA	CVI	0.02	380 (FS)	[27]
	CVI	0.08	272 (TS)	[28]
			356 (FS)	
	CVI	0.1	606 (FS)	[29]
	CVI	0.16	230 (TS)	[30]

TS: Tensile strength; FS: Flexural strength.

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