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Effect of fiber surface state on mechanical properties of C_f/Si–O–C composites

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Abstract

Three-dimensional braided carbon fiber reinforced silicon oxycarbide composites (3D-B C_f/Si–O–C) were fabricated via a polysiloxane infiltration and pyrolysis route. The effects of fiber surface state on microstructure and mechanical properties of C_f/Si–O–C composites were investigated. The change of carbon fiber surface state was achieved via heat treatment in vacuum. The results showed that heat treatment decreased carbon fiber surface activity due to the decrease of the amount of oxygen and nitrogen atoms. The C_f/Si–O–C composites fabricated from the carbon fiber with low surface activity had excellent mechanical properties, which resulted from perfect interfacial bonding and good in situ fiber strength. The flexural strength and fracture toughness of the C_f/Si–O–C composites from the treated fiber were 534 MPa and 23.4 MPa m$^{1/2}$, respectively, which were about 7 and 11 times more than those of the composites from the as-received carbon fiber, respectively.

Keywords: C_f/Si–O–C; Carbon fiber; Heat treatment; Surface state; Mechanical properties

1. Introduction

Continuous carbon fiber reinforced ceramic matrix composites (CFRCMCs), which exhibit high strength, high-fracture toughness, low density, and high-thermal stability, are a most promising candidate materials for utilization at ultra-high temperatures. The precursor-infiltration-pyrolysis (PIP) route for preparation of CFRCMCs has attracted increasing interest in recent years for its advantages such as low processing temperature, controllable ceramic compositions, and near-net-shape technologies. Many different preceramic polymers have been discovered since their original development by Yajima and co-workers [1], such as polycarbosilane (PCS), polysiloxane (PSO), polysilazane (PSZ), which give Si–C, Si–O–C, Si–C–N ceramics after pyrolysis, respectively. Si–O–C ceramics have some attractive physical and thermal properties [2,3]. Moreover, the PSO precursor is commercially available and cheap, whereas PCS or PSZ precursor is expensive and cannot be attained commercially. Thus, PSO precursor derived Si–O–C ceramic and C/Si–O–C composites have attracted increasing research interests [4–7].

The mechanical properties of CFRCMCs are determined by their microstructures, especially the interfacial structures. The category of carbon fibers, the type of precursors and the pyrolysis processing conditions are main factors to the mechanical properties of CFRCMCs. Ma [7,8] has systematically studied the effect of pyrolysis processes on the mechanical properties of C_f/Si–O–C composites fabricated by PIP route, including heating rate, pyrolysis temperature and pyrolysis assisted by hot-pressing. The C_f/Si–O–C composites with flexural strength of 502 MPa and a fracture roughness of 23.7 MPa m$^{1/2}$ were obtained when the first pyrolysis was conducted at 1600 °C with an assisted pressure of 10 MPa. It is well explained the category of carbon fibers affect the mechanical properties of CFRCMCs [9]. Different carbon fibers have distinct surface state, which decides the strength of interfacial bonding between fiber and matrix. Only moderate interfacial bonding can ensure the composites have superior mechanical properties. Some works have been

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carried out to study the effect of the carbon fiber surface state on the mechanical properties of C/C and C/SiC composites [10,11]. So far, there are no reported studies about the effect of fiber surface state on the properties of C_f/Si–O–C composites. It has been proved that the surface state of carbon fiber could be changed by heat treatment [12,13]. So this paper investigated the effect of fiber surface state on the mechanical properties of C/Si–O–C composites fabricated by PIP route and the change of carbon fiber surface state was realized via fiber heat treatment.

2. Experimental

2.1. Sample preparation

The carbon fiber investigated in this study was a PAN-based high strength type with a trade name of T300, manufactured by Toray Co. The physical properties of the fiber were listed in Table 1. Three-dimensional braided (3D-B) T300 carbon fiber preforms with a fiber volume fraction about 45% were used as reinforcements for C_f/Si–O–C composites. The fiber as received commercially was denominated as sample T-0. T-1 was the fiber heated at 1400 °C for 1–2 h and then cooled slowly in a vacuum, which is required to prevent carbon oxidation and its resulting mass loss.

In preparation of C_f/Si–O–C composites, polysiloxane, colorless transparent liquid containing 1.4 wt.% hydrogen, was selected as the precursor to Si–O–C ceramics. Divinylbenzene (DVB), light yellow transparent liquid, acted as cross-linking reagent for PSO. The preparation of three-dimensional braided carbon fiber reinforced silicon oxycarbide composites (3D-B C_f/Si–O–C) included three stages. First, carbon fiber preforms were infiltrated with PSO/DVB solution in vacuum. Second, the preforms filled with precursor were cured at 150 °C for 6 h. Finally, the cured preforms were pyrolyzed at 1200 °C for 30 min in N_2 and atmospheric pressure, with a heating rate of 10–15 °C/min. To get the final composites, the other eight infiltration-cure-pyrolysis cycles were needed. The C_f/Si–O–C composites made with T-0 fiber and T-1 fiber were denoted as T-0C and T-1C, respectively.

2.2. Analysis and measure

The surface chemical state of fiber samples was investigated by X-ray photoelectron spectroscopy (XPS). The data were obtained with an ESCALab220i-XL electron spectrometer from VG Scientific using 300 W Al Kα radiation. The base pressure was about 3×10^{-9} mbar. The survey spectrum was collected from 0 to 1200 eV. The relative amounts of different atoms were estimated from respective areas of assumed Gaussian–Lorentzian curves.

The bulk densities of composites were determined by the water displacement method. Three-point bending tests were used to evaluate flexural strength of composites with the span/height ratio of 15 and a crosshead speed of 0.5 mm/min. The fracture toughness was determined by the single edge notched beam (SENB) method with a crosshead speed of 0.05 mm/min and the span/height ratio of 4. The ratio of notch depth to specimen height was 0.45–0.5. Fracture surfaces of the bending specimens were observed by scanning electron microscope (SEM).

Table 1
Physical properties of T300 carbon fiber

Species	T300
Filament count	3000
Density (g cm^{-3})	1.75
Average diameter (μm)	7
Tensile strength (GPa)	3.45
Young's modulus (GPa)	230
Elongation break (%)	1.5

3. Results and discussion

3.1. Surface state of fiber samples

The surface composition and surface functional groups of carbon fibers can be investigated by XPS. The sizing in T-0's surface had been removed by heat treatment at 400 °C for 1 h in vacuum before the XPS investigation. Fig. 1 showed the spectral features of T-0 and T-1 fibers and the surface composition has been estimated and given in Table 2. The values of binding energy (B.E.) and the atomic concentration (A.C.) were listed for each photo-peak. The two specimens' surfaces were both composed of carbon, oxygen, nitrogen and silicon, but the atomic concentrations had great difference. The carbon atomic percent of T-0 was only 86%, however T-1 had a carbon atomic percent of 98%. The amount of oxygen atom and nitrogen atom in T-0 were nearly eight times higher than those in T-1. The O/C atomic ratio of T-0 was 0.11, which was eight times larger than that of T-1 (0.013). The N/C atomic ratio of T-1 had decreased from 0.038 of T-0 to 0.004 after the T300 fiber was heated at 1400 °C.

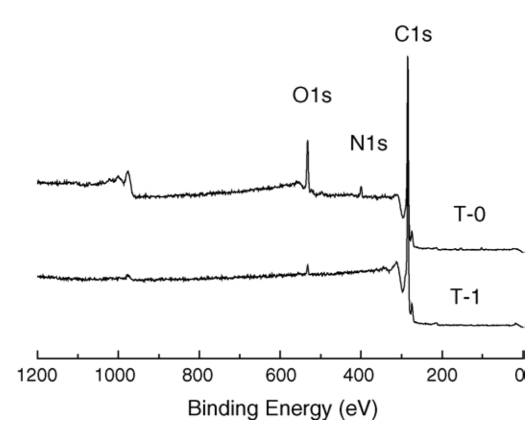

Fig. 1. XPS survey of fiber samples.