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Organic aerogel-impregnated low-density carbon/carbon composites: Preparation, properties and response under simulated atmospheric re-entry conditions

Chonghai Wang, Xiangyu Jin, Haiming Cheng*, Changqing Hong*, Xinghong Zhang

National Key Laboratory of Science and Technology on Advanced Composites in Special Environments, Harbin Institute of Technology, Harbin 150001, PR China

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ABSTRACT

A novel lightweight organic aerogel-impregnated low-density carbon/carbon (C/C) composite was produced through vacuum impregnation using carbon-bonded carbon fibre (CBCF) as substrate and resorcinol-furfural (RF) aerogel as infiltrant. Microstructural analysis showed that fibres in CBCF were uniformly coated with a thin layer of aerogels, acting as the supporting skeletons, to strengthen the aerogel matrix. The as prepared RF-C/C possessed low densities between 0.26 and 0.37 g/cm^3, relatively high compressive strength, ranging from 0.45 to 3.27 MPa, and low thermal conductivities of 0.105 to 0.350 W/(mK) at room temperature. Furthermore, good thermal ablative and insulative properties (recession rates as low as 0.082 mm/s and internal temperature peaks below 90 °C at 38 mm in-depth position as the surface temperature exceeded 2000 °C) under an atmospheric re-entry condition. From mentioned above, RF-C/C present huge application prospects in heat preservation and thermal protection field, especially in energy-saving and aerospace.

1. Introduction

Ablative materials are crucial to many space related applications; these sacrificial materials are used to manage the heat shielding of aerodynamic surfaces, propulsion structures, payloads and ground equipment from the severe effects of very high temperatures and incident heating rates [1–3]. Polymer matrix composites have been widely used in manufacture of ablative materials, owing to their intrinsic advantages such as moderate ablation resistance, good mechanical properties, dimensional stability, lower cost, and higher heat shock resistance [4–6]. However, some issues still remain unsettled, for instance low ablation efficiency, relatively high density and thermal conductivity, owing to the densified structure consisting of fibre reinforcement and polymer matrix. Moreover, there is always an urge to improve the ablation capability of ablative materials to realize thinner and lighter thermal protection structure, thus increasing the payload of the space system [7,8]. Numerous recent investigations have reported to add boron, phosphorus, zirconium, titanium or silicon compounds, graphene or graphene oxide, carbon nanotubes, ceramic particles, nanoclay additives in polymer matrices, or fibre fabrics with altered weave patterns to improve ablation resistance [9–20].

Another newly emerging method is to obtain lightweight ablative materials, to utilize ultra-lightweight additives in polymer matrix, such as hollow silica and/or phenolic microspheres, or to impregnate porous polymer with porous ceramic or carbon fibre felt as substrate and structural support [21–24]. The research on lightweight ablative materials were mainly sponsored by some large institutions and companies, such as National Aeronautics and Space Administration (NASA), European Space Agency (ESA), Applied Research Associates (ARA), the Boeing Company and the Lockheed Martin Company, and they have received plentiful achievements. Fruitful lightweight ablators with fascinating ablation and insulation properties were developed, for instance AVCOAT, MonA, BLA, PhenCarb, SRAM, SLA, PICA, ASTERM, and SIRCA. Moreover, ablation resistance, thermal insulative, thermochemical, and thermophysical properties of these materials were systemically investigated and analysed [25–27]. However, for political and commercial reason, as International Traffic in Arms Regulations and governmental export restrictions, therefore published data of those types of material is limited, and collaborations between different institutions were complicate, or completely restrict, this has to some extent affected the development of this area.

In this context, we proposed and fabricated a novel lightweight carbon/polymer composite by impregnating low-density carbon/carbon (C/C) composite with organic aerogels. Carbon-bonded carbon fibre (CBCF) was used as the structural support and 3D reinforcement, due to its high porosity, low thermal conductivity and high temperature

* Corresponding authors.
E-mail addresses: hmingcheng@yeah.net (H. Cheng), hongcq@hit.edu.cn (C. Hong).

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capability [28–31]. For filler, we select resorcinol-furfural (RF) aerogels based on its easy synthesis and useful properties, such as low density, high porosity, extremely low thermal conductivity, as well as its ability to form a refractory carbon aerogel char and pyrolysis gas during endothermic pyrolysis [32,33]. Synthesis and fabrication was achieved by impregnating CBCF with a sol containing R, F, hexamethylenetetramine (HMTA) and isopropanol (IPA), accompanied by sol-gel polycondensation and ambient pressure drying (APD). Thermal stability, carbonization behaviour, and chemical state evolution of the as-prepared RF aerogels during pyrolysis were analysed. The microstructure of the resulting composite, room-temperature thermal properties and the mechanical properties were systematically investigated. Furthermore, anti-ablation and thermal insulation performance was evaluated under an atmospheric re-entry condition (cold wall heat flux = 1.5 MW/m^2, enthalpy = 20 J/kg and surface pressure = 2.5 kPa) simulated by an arc jet wind tunnel. The developed fabrication method is a facile and versatile strategy to obtain lightweight ablative composites. This detailed mechanical and thermal properties, as well as ablation performance of the lightweight RF-C/C ablative material would be favorable to development of the ablation community.

2. Experimental

2.1. Materials

Resorcinol (R), furfural (F), hexamethylenetetramine (HMTA), and isopropanol (IPA) were purchased from Aladdin Industrial Inc. All chemicals were used as received without any further purification. CBCF was produced by carbonization (in argon atmosphere at 1000 °C for 1 h) of mixtures of chopped rayon based carbon fibre (1.6 mm in length) and equal weight of powdered phenolic resin (200 mesh) attained by pressure filtration technique according to the literature [30]. To investigate the loading level of CBCF affect the properties of RF-C/C composite, CBCF with densities of 0.12, 0.15, 0.18, and 0.21 g/cm^3 were fabricated and named as C/C0.12, C/C0.15, C/C0.18, and C/C0.21, respectively.

2.2. Fabrication of RF-C/C composite

The CBCF composite having dimensions of about 50 × 50 × 25 mm^3 was placed in a vial. The co-precursor solution of R, F, HMTA and IPA with molar ratio R/HMTA = 50 and R/F = 0.5, was added until cover the carbon substrate. Three different solutions with solid contents (ratio of R/IPA) of 0.08 g/ml (sample RF0.08), 0.15 g/ml (sample RF0.15) and 0.20 g/ml (sample RF0.20) were used to synthesize aerogels with different densities. The system was placed in a vacuum container, and the vacuum was maintained until no further air bubbles were formed, to ensure the solution fully infiltrated in the carbon fabric. Then the system was heated to 80 °C in the oven to cure for 7 days. After curing, the artifact was directly dried in air (≤ 30 °C) at ambient pressure until a constant weight was obtained. With the above procedure, RF-C/C composites with low densities (0.26 to 0.37 g/cm^3) were obtained, and they were denoted as RFx-C/Cy, where x is the ratio of R/IPA and y is the density of CBCF, respectively.

2.3. Characterization

The morphologies were observed by using a field emission scanning electron microscope (FE-SEM, FEI HELIOS NanoLab 600i). The Fourier transformed infrared (FTIR) spectra were recorded between 500 and 4000 cm^{-1} from KBr pellets by a Bruker Tensor 27 Spectrophotometer. Thermogravimetric analysis (TGA) was performed using a NETZSCH TG 209C at a heating rate of 10 °C/min from room temperature to 1000 °C with a flow rate of 60 ml/min in an argon atmosphere. N_2 adsorption and desorption isotherms were recorded by a Micromeritics ASAP 2020 V3.00H adsorption apparatus. The specific surface area (S_{BET}) was calculated from the adsorption data in the relative pressure range between 0.05 and 0.35 using the Brunauer-Emmett-Teller (BET) method. The micropore volume (V_{mic}) and micropore surface aera (S_{mic}) were analysed by t-plot theory. The mesopore pore size distribution, mesopore volume (V_{meso}), and mesopore surface aera (S_{meso}) were estimated using Barrett-Joyner-Halenda (BJH) theory, respectively. The BJH analysis was performed from the desorption branch of the isotherms. Before N_2 adsorption, the samples were degassed at 80 °C for 24 h until the mass attained a constant value. Compression testing under ambient conditions was carried out on an Instron 5569 test machine at crosshead speed of 0.5 mm/min using specimens with dimensions of 10 × 10 × 12 mm^3. Thermal conductivity at room temperature was measured by the Hot Disk TPS 2500 thermal constant analyzer at 25 °C using specimens with dimensions of 30 × 30 × 20 mm^3. The thermal ablative properties of the composite were evaluated in arc-jet facility at cold wall heat flux of 1.5 MW/m^2, enthalpy of 20 MJ/kg and surface pressure of 2.5 kPa for 33 s with iso-q specimen [34]. The sample was a cylinder with a spherical nose, the nose radius and the diameter were both 80 mm, and length was 38 mm. Surface and internal temperature were measured by using infrared pyrometer and three K-type thermocouples which were fitted at 18, 28 and 38 mm in-depth from the front sample surface. Additionally, the changes in shape during the testing process were also received and recorded by camera. The recession rate and mass loss ablation rate were calculated by dividing the thickness and mass change pre- and post-test of each specimen into the test time.

3. Results and discussion

3.1. Concept of RF-C/C composite

Fig. 1a describes the synthesis pathway. CBCF composite is a special class of low-density, highly porous C/C composite, has been used extensively in thermal insulation materials, thermal structures and reinforcements of composites [28–31]. The CBCF composites adopted herein have low-density of 0.12 to 0.21 g/cm^3 and high porosity of 87% to 93%. They were produced from low thermal conductivity and excellent refractory rayon based carbon fibres of nominal length 1.6 mm and diameter 5–7 μm [30]. The sol-gel polycondensation after vacuum impregnation converts the RF sol to wet RF aerogel, and the APD removes the pore-filling solvent and residual chemicals to give a RF-C/C composite that has a relatively low density of 0.26–0.37 g/cm^3 and high porosity of 76 to 81%. Microstructure of the CBCF in Fig. 1b shows that the chopped carbon fibres are bonded together at the intersections of adjacent fibres with pyrolytic carbon. Noticeably, a 2D planar random structure was obtained as the carbon fibres in the xy direction are distributed homogeneously in plane, while in the z direction the fibres are uniformly distributed over the height of the composite. Therefore, both the properties of the CBCF and RF-C/C composite are isotropic within the xy plane but anisotropic behaviour is observed at angles to this plane. From the micrographs of the RF-C/C composite (Fig. 1c), it is obvious that the RF aerogels uniformly coat the surface of fibres and maintain a homogeneous porous aerogel structure and high porosity in the RF-C/C composite. The CBCF acted as a supporting skeleton that could increase the mechanical properties of RF aerogels.

3.2. Microstructure, textural properties and thermal stability of RF aerogels

FE-SEM and N_2 adsorption measurements were performed to examine the porosity microstructure of the RF aerogels with different solids contents. The results are shown in Fig. 2 and Table S2. The as-prepared RF aerogels display the nano-particle structure analogous to that of the typical aerogels prepared by Pekala [35]. One could see that the aerogel network of the RF aerogels was composed of grape-like aggregates of primary nano-particles, which are connected into a bulk network. There are many nano-scaled interconnected pores among the

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