



The degradation behavior of C/C composites in high-energy atomic oxygen



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ABSTRACT

In low earth orbit (LEO), spacecraft, space structure and other applications encounter a degradation problem induced by the impact of atomic oxygen (AO) in the space environment. C/C composites (C/Cs), which are widely applied to different applications in space systems, were tested to study the irradiation and bombardment effects of LEO space environment on the composites. The degradation was analyzed in the aspect of mass loss, morphology and flexural strength and thermal conductivity properties. Results show that the specimens are bombarded by high-speed AO with high activity during AO irradiation test. The flexural strength of C/C composites after AO irradiation test for 24 h (AO24, 150.4 MPa) is decreased by 25.8% than C/Cs (202.7 MPa). The coefficients of thermal conductivity of C/Cs after AO irradiation show an increase. Carbon fibers and pyrolytic carbon (PyC) were also exposed to AO irradiation test. Results show that the denudation rate of PyC is slower than that of C/C composites and carbon fiber. Besides, the denudation of carbon fiber (form crisscrossed ravine) is most serious. PyC shows the least damage. This work may provide not only the influence of AO irradiation on C/Cs but the protection of C/Cs from AO irradiation.

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1. Introduction

Carbon fiber reinforced carbon matrix composites (C/C composites) have attracted a lot of attention in recent years by virtue of their unique properties [1,2], such as low density, high melting point, low coefficient of thermal expansion (CTE), retention of mechanical properties at high temperature [3–6]. These superior features make C/C composites ideal materials in ultra-high temperature environment, such as spacecraft, space structure and other applications [7–14]. However, spacecraft, space structure and other applications experience irradiation and bombardment of atomic oxygen (AO), which are primary component of the atmosphere in the low earth orbit (LEO). AO originates from earth's atmosphere, in which oxygen molecules are disintegrated into neutral atoms by ultraviolet rays and the formed AO remains at LEO altitude [15,16]. AO is the major constituent in low Earth orbit (LEO). The content is about 80%. Due to the spacecraft orbital

velocity is approximately 8 km/s, AO has the sufficiently high flux (approximately 10^{14} – 10^{15} atoms/cm²/s) and high translational kinetic energy (about 5 eV) [17,18]. Raja Reddy [19] performed the actual flight experiments in LEO, which were conducted by lots of materials on several space shuttles and long duration exposure facility (LDEF). These experiments provided lots of useful practical information on the damage in the real flight. However, sometimes it is difficult to control the necessary parameters. The experimental simulations on the ground have also been performed using various techniques of accelerating AO. Koji Fujimoto et al. [20] investigated an experiment on the ground for the degradation of carbon-based materials. The mass loss, eroded surfaces and surface profiles of the carbon-based materials were investigated. In Ref. [21] performed by Arnold et al. the reaction rate of amorphous carbon with AO was investigated. Joo-Hyun Han et al. [22] investigated the low earth orbit space environment simulation and its effects on graphite/epoxy composites. Misak et al. [23] studied the thermal fatigue and hypothermal AO exposure behavior of carbon nanotube wire. Although there are several researches on the affection of graphite or monolithic carbon affected by AO, limited researches are reported on the AO irradiation and bombardment effect on carbon

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fibers, pyrolytic carbon (PyC) or C/C composites. C/C composites are considered as ideal structural materials capable of applications in ultra-high temperature environments [24–26]. This paper studies the irradiation and bombardment effect on the surface morphology, mechanical properties and thermal properties of C/C composites. To have a better understand of the affection of AO irradiation and bombardment, carbon fibers and PyC are also exposed to AO irradiation test. The mass losses were measured to observe the denudation of materials after exposure different time. 3D confocal laser microscope was used to analyze the surface roughness of the specimens. Scanning electron micrographs (SEM) were used to observe the eroded surface morphology. Crystalline phase of the prepared specimens before and after AO irradiation test were identified by X-ray diffraction (XRD). Three-point bending tests were performed to examine the effect of AO irradiation and bombardment on mechanical properties. A thermal properties analyzer was used to measure the specific heat capacity of the specimen.

2. Experimental

2.1. Specimen preparation

To obtain C/C composites, 2.5 dimensional (2.5 D) needle-punched carbon fiber felts with a density of 0.45 g/cm^3 (Yi xing Tian niao High Technology Co. Ltd., China) were used as preforms. Carbon fibers were commercial 12 k T700 PAN-based (Toray, Japan) with a filament diameter of 7 mm, tensile strength of 4.9 GPa and fiber volume fraction of the preform was about 25%. Schematic diagram of C/C composites is shown in Fig. 1. It was fabricated by repeatedly stacked layers of 0 non-woven carbon fiber cloth, short-cut fiber web, 90 non-woven carbon fiber cloths and short-cut fiber web with needlepunching in Z direction step-by-step [27,28]. Thermal gradient chemical vapor infiltration (TCVI) was employed to infiltrate PyC into preforms to obtain C/C composites with a density of about 1.76 g/cm^3 . The density was measured according to Archimedes law. This method is based on dipping specimen buoyancy, which can evaluate the porosity and density by measuring the amount of fluid absorbed by the specimen [29]. During the TCVI procedure, natural gas with a flow rate of 15–20 L/min was used as carbon source. The deposition pressure, temperature and time were 5–10 kPa, 1273–1473 K and 260 h, respectively.

Finally, the C/C composites were heat-treated at 2273–2573 K for 2 h. To obtain PyC, needle-punched carbon cloth (5 layers, 3 k) was also put into the furnace to use thermal gradient chemical vapor infiltration (TCVI) to infiltrate PyC.

Rectangular specimens ($55 \times 10 \times 4 \text{ mm}$) and disk specimens ($\phi 12.65 \text{ mm} \times 3 \text{ mm}$ and $\phi 30 \text{ mm} \times 8 \text{ mm}$) were cut from the prepared C/C composites and lightly hand-abraded with 80 and 300 grit SiC papers. Then they were cleaned in ultrasonic devices with ethanol and dried at 373 K for 12 h for test.

To have a better understand of the affection of AO irradiation and bombardment, carbon fibers (carbon cloth: $10 \times 10 \text{ mm}$) and PyC ($10 \times 10 \times 10 \text{ mm}$) are also exposed to AO irradiation test.

2.2. AO irradiation test

The AO irradiation test was measured in a coaxial source AO ground simulation test device (Fig. 2), which is composed of microwave generator, magnet coil, vacuum pump, neutralized plate and IPC system [22]. The device can generate AO flux through weakly ionized remote oxygen plasma with a radio-frequency (RF, 2.45 MHz) plasma source. The power supply of the system is 600–1500 W and the gas supplies are O_2 and Ar. In plasma chamber is vacuum condition, which is generated by pumping system. The estimated AO flux onto the target specimen surface was of the order of $1.0 \times 10^{15} \text{ atoms/cm}^2/\text{s}$. When exposed to AO irradiation test, Kapton can be seriously eroded by AO with a well-known erosion yield of $3.0 \times 10^{-24} \text{ cm}^3/\text{atom}$ [30]. Thus, Kapton is used as the standard material for the estimation of the AO flux. The value of AO flux is based on the witness sample erosion of Kapton HN. The method to estimate AO flux can be described as this: Firstly, overlap two Kapton HN specimens. Then the specimen (test specimen) was exposed to AO in the beam. The other specimen (reference specimen) was put into vacuum environment and at the same temperature, but without AO bombardment. After bombardment, measure the mass of the two specimens (marked as m_t and m_r). The mass loss caused by AO effect (marked as Δm) can be calculated by the following equation (eq. (1)):

$$\Delta m = m_t - m_r \quad (1)$$

where m_t and m_r are the mass loss of the test specimen and reference specimen, respectively.

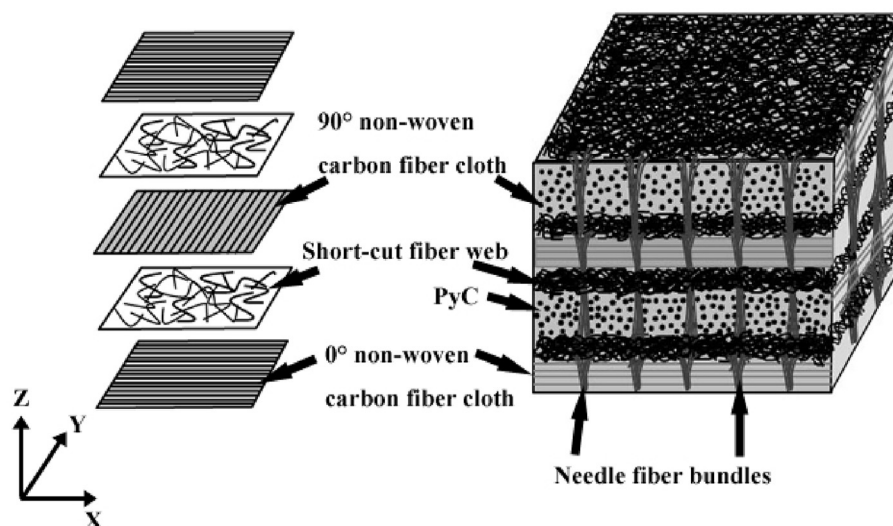


Fig. 1. Schematic diagram of the architecture of C/C composites.

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