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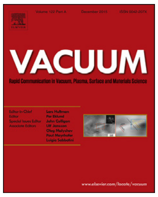

Effect of vacuum thermal cyclic exposures on the carbon/carbon composites

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ABSTRACT

The study aims to investigate the effects of vacuum thermal cycling on carbon/carbon (C/C) composites used in aerospace under simulated low earth orbit (LEO) environmental conditions. The C/C composites were tested at the temperature range from −120 °C to 120 °C for 100 cycles under the high-vacuum state of 1.3x10^{-3}Pa, the composites were characterized through the assessment of the thermal physical and mechanical property changes. It follows from the experiment that the cyclic thermal stress derived from thermal cycling could influence the stability of C/C composites which result in an abnormal phenomenon of thermal expansion of C/C composites under cryogenic temperature. The flexural strength initially decreases from 105.51 MPa to 74.83 MPa with the thermal cycles increasing to 30, then increases to 96.41 MPa after 100 thermal cycles, showing a recovery tendency (28.84%) of flexural strength compared with 30 thermal cycles and maintains the initial flexural strength of 91.39%.The implications of these processes based on the relation between coefficients of C/C composites between vacuum thermal cycling were discussed and an interfacial damage model based on microscopic observation was proposed.

1. Introduction

Carbon fiber reinforced carbon composites (C/C composites) display superior mechanical properties and structural integrity at temperature up to 3000 °C either in vacuum or in inert environment [1,2]. The strength of C/C composites could increase at 2000 °C or higher, which has made the material ideal candidates for structural materials in aerospace applications [3]. However, in space environment, space crafts are subjected to high vacuum, thermal cycles, high energy charged particles (protons and electrons), atomic oxygen (AO) and some other harsh environmental damage, which could lead to the deterioration in properties of the composites [4,5]. The typical and cannot be neglected environmental effect of space is thermal cycling at low earth orbit (LEO, between 100 km and 1500 km above the Earth's surface) where the aircrafts, and wing leading edges of space shuttles pass in and out of the earth's shadow repeatedly. The composites have to undergo the sharp temperature changes when it is in space environment, which could lead to the degradation of mechanical properties of composites [6–8].

Considering the importance of the vacuum thermal cycling, several studies [6,7] [9–11]have been conducted. Gao et al. [6], Shin [7] and Funk and Sykes [11] have particularly focused on the understanding of cumulative damage development due to the cyclic exposure. Shin et al. [7] has examined the effects of thermal cycling environment on the graphite/epoxy composites materials. It reported that the matrix dominated mechanical properties such as transverse flexural strength showed a sharp reduction after thermal cycling. Rouquie et al. [12] has studied thermal cycling of carbon/epoxy laminates in neutral and oxidative environments. The observations showed that cyclic thermal stresses cause an acceleration of matrix cracking process. However, there are not many related reports about the cryogenic response of carbon fiber reinforced carbon composites induced by periodic shadow of the Earth. In order to improve the orbital operation and reusability of spacecrafts, it is essential to investigate the performance of C/C composites and predict the design margin under vacuum thermal cycling environment.

The main purpose of this study is to understand the effects of different vacuum thermal cycling on the thermal physical and mechanical properties of C/C composites. Vacuum thermal cycling tests were conducted up to 100 cycles on the C/C composites with

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the purpose of research C/C composites degradation and its reliability in maintaining their outstanding performance against the harsh space environment. An interfacial model based on the influence of cyclic thermal stress induced by thermal cycles is proposed to interpret the flexural fracture behavior of the C/C composites.

2. Experimental details

2.1. Materials

Needle-punched integrated felts made up of T-300PAN Fibers were used as preforms (Fig. 1). In Fig. 1, carbon fiber felts have been laminated, and two successive layers have been oriented at an angle of 90. Then they were needled progressively till the thickness of preforms. The preforms were densified to a density of 1.65–1.70 g/cm^3 by isothermal isobaric chemical vapor infiltration (ICVI) process. C/C composites were observed under polarized light microscope (PLM, Leica DMLP optical microscope). Fig. 2 showed PLM micrograph of cross section(X–Z) of the as-prepared C/C composites. The image indicated that carbon matrix of the composites exhibited smooth laminar pyro-carbon (ML, middle texted pyro-carbon).

2.2. Vacuum thermal cycling simulated system

Vacuum thermal cycling experiments were carried out in a ESS-KWGA1507 type thermal cycling machine, produced by GALAXY Equipment Company of Co. LTD. in Chongqing, China. To determine the minimum hold time, it was used a 0.201 mm thick k-type thermocouple bonded between a specimen of 4 mm × 10 mm × 50 mm to measure temperature. The time necessary for the center ply of this sample to reach the required temperatures (within±4 °C) was recorded when the samples were placed into cryogenic/elevated cycle experiment box. In this study, one thermal cycle was defined as a change in temperature from room temperature to −120 °C, then up to +120 °C and back to room temperature again, with a brief hold time about 30 min at either peak temperature +120 °C, −120 °C to attain thermal equilibrium. The chamber pressure was lower than 1.3×10^{-3} Pa, and no loads were applied to the samples. Heating and cooling of samples were achieved by using three 1500 W heating pipes and a copper cooling coil fed with liquid nitrogen (LN$_2$), respectively.

2.3. Measurement

The mass loss of C/C composites under various thermal cycles was measured. The changes in linear coefficient of thermal expansion (CTE) were measured in directions perpendicular (Z direction) and parallel (XY direction) to the carbon fiber felts direction, respectively. Raman micro-spectroscope (Renishaw in Via) is used to observe the microstructural changes of the fibers and the pyrolytic carbon matrix and characterize the local stress caused by different thermal cycles [13,14]. The three-point bending tests were carried out on an electrohydraulic-servo testing machine (Instron Co. Ltd., 8872) with a 40 mm span and a crosshead speed of 0.5 mm/min [15]. Five rectangular bars of 4 mm × 10 mm × 50 mm were tested to evaluate mechanical properties of the composites and were carried out in accordance with Q/GB 95-92 [16]. The morphology of the fractured surface of the samples after three-point bending test was observed using a ZEISS SUPRA scanning electron microscope (SEM).

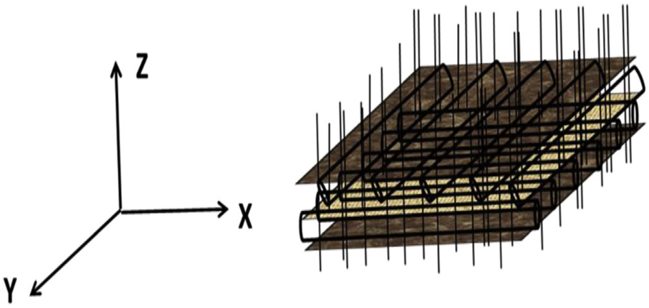

Fig. 1. Needle-punched integrated preforms of C/C composites.

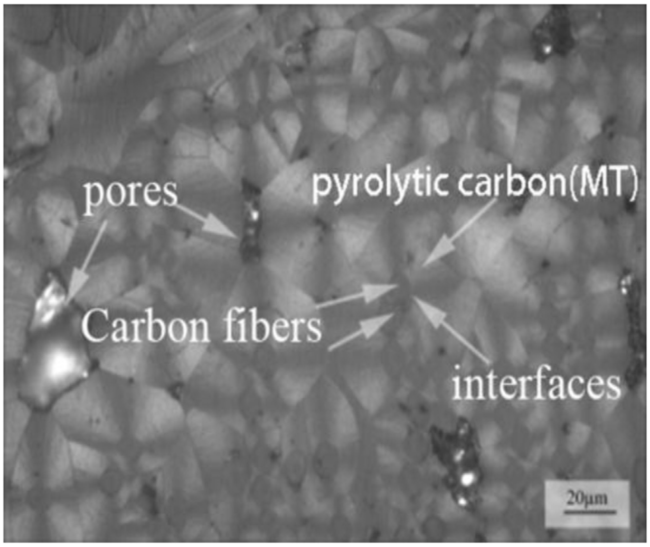

Fig. 2. PLM micrograph of C/C composites.

3. Results and discussion

3.1. Changes in mass loss

Fig. 3 shows the mass loss ratio of the C/C composites subjected to various thermal cycles. With the increasing thermal

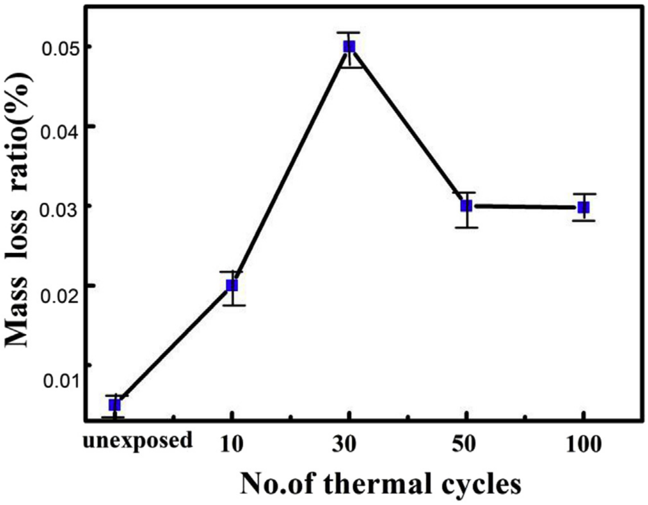

Fig. 3. Mass loss ratio of the carbon/carbon composites subjected to different thermal cycles.