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Test methods for investigation of reusable launch vehicle materials under severe environment

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Abstract. Ceramic matrix composite (CMC) integrated with various advanced properties is a promising material in many space projects. In those applications, CMC will be subjected to various extreme environments with temperature range of −170 to 2000 °C as well as space particles. A series of methods have been developed to simulate different stage of severe environment, i.e., launching stage, staying stage and re-entry stage. C/SiC related materials have been investigated by these simulation methods. A lot of useful information obtained by the series of simulation methods have been remarkably enhanced our understanding on the behavior of CMC used for RLV hot structures.

1. Introduction

Ceramic matrix composite (CMC) integrated with various advanced properties is one of cornerstone for Crew Explore Vehicle (CEV), which will replace the space shuttle in ferrying crews to and from the space station and will enable to the Moon, Mars, and beyond in future [1]. The use of load carrying low-mass and high temperature stable ceramic matrix composite (CMC) structures, such as the carbon fiber-reinforced silicon carbide ceramic matrix (C/SiC) composites, is the best choice in order to meet this requirement [2–5]. Many projects competed in the CEV or the next generation hypersonic space vehicles emphasize studies on practical C/SiC elements, in particular for airframe structures, hot and integrated structures, thermal protection system and leading edge structures, etc. [6].

In those applications, CMC will be subjected to various extreme environments with temperature range of −170 to 2000 °C [7, 8] as well as molecular oxygen (MO), molecular water and atomic oxygen (AO) [9, 10] depending on the flight stage. Oxidation by MO is the major problem during launching, however, oxidation by AO is the main issue as well as cryogenic temperature during staying in low earth orbit (LEO). During re-entry, large amounts of energy are set free near the surface due to the high entry velocity. The increasing oxygen content in the atmosphere emphasizes the oxidation effect.

Besides the high risk and cost, the real flight testing cannot offer enough information for the degradation of CMC. Therefore, as real flight conditions are difficult to be achieved due to high cost,

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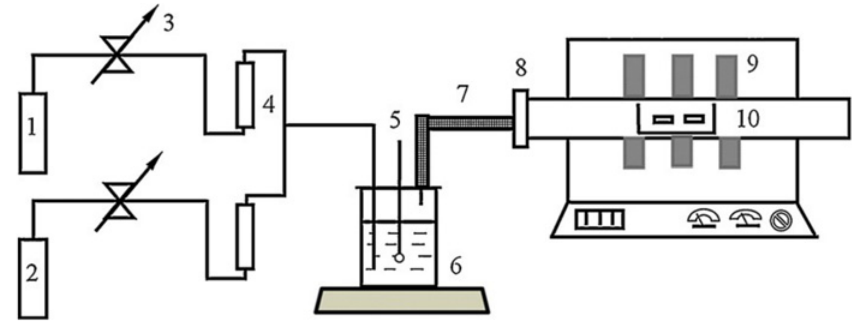

Figure 1. Schematic diagram of the oxidation experiment set. (1) Oxygen, (2) nitrogen, (3) valve, (4) flow meter, (5) thermometer, (6) water bath, (7) heater band, (8) seal ring, (9) heating unit and (10) specimens.

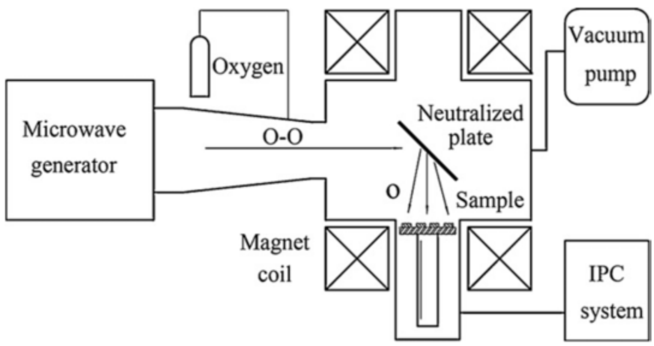

Figure 2. Coaxial source atomic oxygen ground-based simulation facility.

ground test facilities are used to qualify the necessary heat-protection materials. It is difficult and unnecessary to realize all factors in one testing. A series of methods have been developed to simulate different stage of serve environment.

2. Experiment simulation of launching stage [11]

Depending on the flight task, CMC used as hot structure parts will be attacked by MO, AO or both, respectively. Most studies interested in solely MO oxidation for C/SiC composites, their AO effect has little information from previous reports; the coupled oxidation by MO and AO is beyond understanding even more. Therefore, it is essential to investigate the C/SiC behaviors under the above corresponding environments for assessing its durability or predicting their design margins.

In the MO oxidation, the as received C/SiC samples were exposed in a simulated air (O2, 21 vol%) at 1500 °C for 10 h. The testing system is shown in Fig. 1.

For the AO oxidation, the specimens were exposed in a ground simulated AO atmosphere (Fig. 2). When the inner pressure is reduced to 4.0×10^{-3} Pa by a vacuum system, the energy of 2.4 GHz microwave discharge is coupled and introduced upon pure oxygen molecules through an antenna transmission system, generating high-density oxygen plasma. The plasmas are accelerated to gain enough energy through a magnetic field, and subsequently dash against a biased plate, in which the electrons were absorbed in the flux and neutralized the plasma particles creating neutral AOs. Meanwhile, the AOs are reflected upon the plate, and veered to target materials. The energy and flux of AO can be adjusted by microwave power and the magnetic field. Their fluxes and flux distributions are

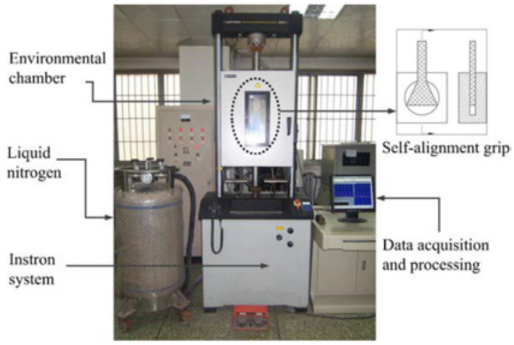

Figure 3. The cryogenic experimental system.

measured using a silver oxide-quartz catalytic probe, and the AO energy is determined by a quadrupole mass spectrometer. The average energy of AOs is 8.4 eV, corresponding to that of natural AO in LEO [12, 13].

In coupled oxidation, the mode A (first MO and then AO) and the mode B (first AO and then MO) oxidation were performed in high temperature furnace and the AO facility respectively. The specimen surfaces were observed by SEM (S-2700, Hitachi High Technologies Corporation, Japan) and their chemical states were investigated by XRD (D/max-2400, Rigaku Co., Japan) and XPS (Axis Ultra, Kratos Analytical Ltd., UK).

In the MO oxidation, an inert surface is formed on SiC. At the AO oxidation, the Si of SiC is preferentially etched off leaving a certain depth of C layer on the sample. Two types of coupled oxidations defined as mode A (first MO and then AO oxidation), and mode B oxidation (first AO and then MO oxidation) were investigated systematically in this study, and found that the prior oxidative effect majorly determined the corresponding coupled oxidation behavior. From microscopic observation and fractural test, the mode B oxidation induced a more serious degradation upon the material than that of the mode A.

3. Experiment simulation of staying in LEO [14–18]

During staying in LEO, CMC's cryogenic respond induced by periodic shadow of the Earth, has been paid little attention. Bombed by space particles such as AO, proton and electron is another important problem affecting properties of CMC. In order to improve space-craft's orbital operation and reusability, it is essential to investigate the material performance and predict its design margin under cryogenic environment or space particles environment.

An Instron fatigue testing system (Model 8801, Instron Ltd., High Wycombe, UK) and an Instron static axial extensometer (Model 2630-105, Instron Ltd., High Wycombe, UK) were used to investigate the composite tensile performance. As shown in Fig. 3, an environmental chamber integrated with the Instron-8801 was used to get the desired temperatures, which is controlled and measured by a Eurotherm PID controller (Model 2408, Eurotherm GmbH, Vienna, AT), using standard type K thermocouples placed within the chamber. For accurate measurement, a pair of self-alignment grips was designed for the as received specimen. In this study, the cryogenic test was performed in nitrogen atmosphere; the room temperature (RT) investigation was carried out in ambient atmosphere. Actually, the RT effect is considered as reference in this study, acting as a logical start to analysis cryogenic property.

Liquid nitrogen evaporation was used to get cryogenic temperatures. The pairs of self-alignment grips held the specimen are set free to compensate systemic shrinkage, while a routine temperature

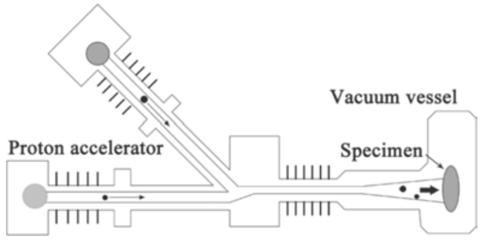

Figure 4. Ground based space radiation environment simulator.

decline is being performed. At the target temperature, the step is to balance thermal state about 30 min, guaranteeing temperature fluctuation below 1 °C. Specimens were then tensioned at 25, −40, −80 and −100 °C with constant loading rate of 0.05 mm/min, and at least 10 specimens were tested at one temperature point.

AO was obtained by using the same system shown in Fig. 2. Proton and electron was got by a ground based space environment simulator (Lanzhou Institute of Physics, Lanzhou, China), the schematic diagram showed in the Figure 4. The materials were irradiated by 0.1 MeV proton with a flux of 7.75×10^{10} p/cm^2 · sec.

In cryogenic environment, the 3D C/SiC composite has a considerable ductility and characters the behavior of non-sudden disruption. The CTE mismatch between carbon fiber and SiC matrix is considered as the profound impact on the initiation and evolution of the damage at cryogenic temperatures. The specimen suffers the most violent degradation at −40 °C due to the debonding of the interface between carbon fiber and SiC matrix. When temperature declines to −100 °C, the mechanical properties are restored well because of the rebuilding of the interface. Increasing the volume fraction of carbon fiber along the tensile load direction is potential to enhance its cryogenic stability. Decreasing the thickness of the PyC interlayer among the carbon fiber filaments can increase the interfacial frictional force, but increasing the thickness shall benefit the crack deflexion within the single fiber bundle. The short carbon fiber reinforced C/SiC composite serving as a load carried structure exhibits no advantage in cryogenic environment.

In space particles environment, AO is considered as the most erosive particle to spacecraft materials in LEO. Carbon fiber, SiC fiber, carbon/carbon (C/C), modified C/C composites and SiC coated C/SiC were exposed to a simulated AO environment. The carbon fiber and the C/C specimens undergo significant degradation under the AO bombing, while the erosion rate of SiC-coated C/SiC is about 50 times lower. Evidences show that Si element is preferentially etched from the SiC lattice, so amorphous carbon and diamond-like carbon is periodically generated on the tested composite surface. Statistical analysis shows that the C/SiC specimens have no significant change in flexural properties after 1-year equivalent AO treatment. According to the effects of AO on C/C-SiC and SiC-coated C/C, a condensed CVD-SiC coat is a feasible approach to protect C/C composites from AO degradation.

Carbon fiber, SiC fiber, C/SiC, and SiC/SiC were also irradiated in 0.1 MeV proton and 0.1 MeV electron. The strength of the carbon fiber decays obviously, and the strength data tends to concentrate with the irradiation time increasing. The strength data of SiC fiber becomes scatter after 4 hours proton treatment, but the data becomes concentrate and the strength simultaneously decreases significantly after 8 hours. The combined irradiation by proton and electron has a similar effect to the individual proton irradiation. The proton irradiation seems more serious than the combined irradiation from the view of the fiber strength degradation. The interaction between the outside electrons of the target atom (C or SiC) and the proton (or electron) particles is the primary irradiation damage mechanism for SiC-CMCs, and the depth of the incident proton is about 1 μm in C or SiC according to SRIM calculation. The results

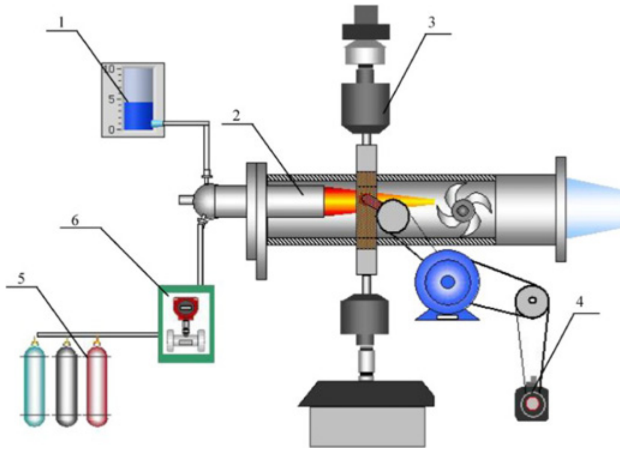

Figure 5. The schematic diagram of the experimental simulation method. 1, water-cooling system; 2, atmospheric subsonic gas-fired wind tunnel; 3, loading system for mechanical property tests; 4, servo-actuator of the rotating system; 5, gas supply; 6, mass flow controller of gas.

show that the proton and electron irradiation of 0.1 MeV has little influence upon C/SiC and SiC/SiC composite.

4. Experiment simulation of re-entry [19–22]

During re-entry, large amounts of energy are set free near the surface due to the high entry velocity. Besides the high thermally and mechanically loading effects, the high-temperature joining strength and especially the high temperature tribological behaviours are becoming new important issues for some hot structures, such as leading edge segments, rudders, nose cap, and chin panel. What more, all the above problems should be simultaneously considered in the hinge bearing of the body flaps.

Thereby, a suitable simulation method should create a high enthalpy thermal environment with an oxidizing atmosphere and a stress condition as well as a tribological accessory for the friction behavior of a C/SiC hinge. Figure 5 displays the schematic diagram of the present experimental simulation method for performance research of CMC during re-entry. This simulation method was realized by two major simulations, thermal environment, and coupling stress environment. The high-temperature gas combustion wind tunnel simulates the thermal environment. The gas-flow controller realizes the oxidizing atmosphere with variable partial pressure of oxygen and water vapor. The combined mechanical loading system and rotating servo driver simulate the coupling loading/stress environment.

The high enthalpy thermal environment with different temperatures and partial pressures of oxygen were finally realized by changing the flow ratio and mixture ratio of oxygen, nitrogen, and methane. The temperature can be adjusted in the range of 600–2000 °C by a step of 50 °C, and the mass content of oxygen can be changed in the range of 10–30% by a step of 5%. The partial pressure of oxygen can be adjusted in the range of 10–30 kPa, and the partial pressure of water can be changed in the range of 30–60%. The combustion gas flow speed is about 20 m/s, the temperature variance is 72–4 °C, the maximum uniform heating cross section is about Ø 60 mm and the maximum duration for each combustion is 1800 s.

Tensile behavior in a simulated re-entry environment was studied and compared with that in vacuum. A significant strength reduction was observed in re-entry environments due to the interaction of load, high temperature, and oxidation. The non-uniform oxidation of fibers weakened the load-carrying ability due to decreasing effective loaded area and hence strength. Finally, a minimum diameter of total fiber cluster embedded in composites is suggested about 220 μm for use under high load in re-entry environments at 1800 °C.

Stress-oxidation was investigated under different stress levels from 0 to 200 MPa up to 1800 °C. The friction behavior of the hinge bearing system was studied under high loads (up to 25 kN) and low rotating velocities. The mechanical properties of the bolts with a thread connection were conducted under tensile and shear fatigue at both room temperature and elevated temperature. The results show that the stress-oxidation behavior of 2D-C/SiC composites in a combustion environment is a combined effect of extremely high load, high temperature, and oxidation. The load and temperature influenced the crack openings and thus the oxidation of carbon fibers in the precracked composites. The combustion environment mainly determined the time to failure of the specimens by oxidation damage under a high applied stress. Reliable thermal load-carrying ability and stable friction performance of the hinge bearing is demonstrated in high-temperature combustion environments with extremely high loads. The oxidation products of SiO_2 at high temperatures between surfaces played an important role to modify the friction by providing a protective layer. The room temperature tensile and shear strength of the bolts made of needled C/SiC are 139 MPa and 83 MPa, respectively. Even at 1800 °C in a combustion environment, the strengths still retained about 116 MPa with a maximum decrease of 13%. More importantly, the bolts did not suffer significant mechanical degradation after tension-tension fatigue at 1 Hz for 24 h.

5. Outlook

A lot of useful information obtained by the series of simulation methods have been remarkably enhanced our understanding on the behaviors of CMC used for RLV hot structures. However, there is still a lot of issues need to be studied for the modification of CMC and safe use of CMC parts, for example, how the damage happened in the former stage effects the damage evolvement in the next following stage.

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