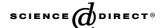


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Low earth orbit space environment simulation and its effects on graphite/epoxy composites

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Abstract

In this study, a low Earth orbit (LEO) space environment simulation facility, capable of simulating major LEO space environment constituents such as high vacuum, ultraviolet (UV) radiation, thermal cycling and atomic oxygen (AO) atmosphere, is introduced. Under the simulated LEO space environment, graphite/epoxy composites, which are widely applied to space structures, were tested to study the LEO space environment effects on the composites. Tensile properties, as well as mass loss of the graphite/epoxy composites, after being exposed to AO atmosphere and the synergistic LEO space environment, were investigated. The surface morphology of the composites under LEO space environment effects was also observed by scanning electron microscope (SEM). Experimental results showed that LEO environment and its synergistic effects cause considerable damage to the surface of composites. © 2004 Elsevier Ltd. All rights reserved.

Keywords: Space environment; Atomic oxygen (AO); Graphite/epoxy composite; Space structure

1. Introduction

Composite materials are widely applied to space structures and systems on account of their extraordinary optical, thermal, electrical, and mechanical characteristics. For instance, composite materials with high specific stiffness and strength, excellent fatigue resistance, and low coefficient of thermal expansion, applied to the space structures, provide optimal conditions that meet the structural design requirements of spacecrafts, space shuttle, space stations, and launch vehicles. In addition, using composite materials to design and manufacture the space structural components results in the reduction of structure assembly time and overall costs. Despite these positive aspects of utilizing composite materials, if the structures are required to operate for an extended period of time in space, they may have difficulty in

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maintaining their outstanding performance against the harsh space environment.

The space environment, especially low Earth orbit (LEO), where a number of spacecrafts, including Space Shuttle and International Space Station operate, is very hazardous to polymer matrix composite materials. The LEO space environment constituents consisting of high vacuum, ultraviolet (UV) radiation, thermal cycles, atomic oxygen (AO), charged particles, electromagnetic radiation, micrometeoroids, and man-made debris, significantly degrade the material characteristics of polymers and polymer matrix composite (PMC) materials. Being exposed to the severe LEO space environment, the composite materials used in space structural components will undergo fatigue cracking caused by thermal cycling, surface erosion by AO attack, structural modification and mass loss by outgassing, modification of material properties by UV radiation, delamination by collisions with micrometeoroids and man-made debris with high velocities. Thus, in designing the space structures using composite materials, possible deterioration

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of mechanical properties caused by long-term exposure to the LEO space environment must be carefully considered. Consequently, reliable understanding of the LEO space environment effects on composite materials, as well as the investigation of the LEO space environment characteristics are crucial.

To date, a number of studies on space environment effects on composite structures have been conducted to understand the modification mechanisms of material properties after being exposed to the space environment constituents and to select appropriate materials for the space composite structures. There are two different methods to perform the space environment exposure experiment to test material characteristics under the space environment: in-flight experiment and accelerated ground simulation experiment. In-flight experiment offers accurate and reliable information about the degradation of material properties under space environment, since material samples carried on space stations or on spacecrafts can be exposed to the real synergistic space environment effects. The accelerated ground simulation experiment, on the other hand, offers the unlimited number of testing materials, as well as the reduction of cost and time consumption. Both the inflight and ground simulation space environment exposure experiments have been used to provide valuable information to design and manufacture the space structures and/or structural components using composite materials.

In this study, a ground facility for simulating the LEO space environment, designed and developed in Smart Structures and Composite Materials Laboratory at Korea Advanced Institute of Science and Technology (KAIST), is introduced. And the LEO space environment effects on typical graphite/epoxy composites are presented and understood by investigating their LEO space environment characteristics—that is the changes in tensile properties, mass, and surface after the LEO space environment exposure.

2. LEO space environment simulation

2.1. LEO space environment

Orbiting the Earth, spacecrafts encounter very different environment with the one we experience on Earth. They face space environmental hazards. The hazards are composed of a variety of atomic species, charged particles, electro-magnetic radiation, micrometeoroids and space debris, depending on extremely low pressure, high thermal change and solar activity in space. If any of these hazards contact the spacecraft, they may harm the spacecraft enough to bring it to an unexpected end of spacecraft's mission, caused by failure of engineering subsystem or premature reentry into the atmosphere [1].

2.1.1. High vacuum

Pressure in space is defined as ultra-high vacuum of less than the order of 10^{-5} Torr. The magnitude of pressure is dependent on altitude and solar effect so as to be on the order of 10^{-5} Torr at an altitude of 200 km and 10⁻¹² Torr at 6500 km. Under ultra-high vacuum condition, a process called outgassing occurs in polymeric materials, which consequently induces loss of dimensional stability of materials, distortion of structures, contamination of surface of material, and unfavorable effects on material properties. Outgassed material is usually composed of moisture, catalyst and monomer. If, for example, 100 nm thickness of the outgassed material is dashed against a mirror or thermal control surface, its absorptance and transmittance deteriorate severely. Hence, the use of low outgassing materials is essential for designing spacecraft carrying sensitive optical payloads [1].

2.1.2. Ultraviolet (UV) radiation

UV solar rays with wavelengths ranging from 100 to 200 nm are absorbed by atmosphere of the Earth [2]. Orbiting the Earth's atmosphere, spacecraft is exposed to the UV radiation, energy which is enough to break molecular bonds in polymer such as C–C, C–O and functional group, build volatile fragments, and alter materials properties of polymer by scissioning or cross-linking [3]. Consequently, degradation of thermo-optical and mechanical properties of structural component, as well as subsystems, can take place.

2.1.3. Thermal cycling

During the operation, spacecraft orbiting the Earth experiences severe thermal difference between the surface exposed to solar rays and the surface unexposed. It also experiences difference between day, having maximum solar activity, and night, having no solar activity. The thermal cycling generates a mismatch in the coefficients of thermal expansion (CTE's) of the matrix and fibers, hence initiating microcracks in matrices of composite materials, which results in degradation of mechanical properties of the composite materials. Moreover, exposed to thermal cycling, fracture of structural components is induced by thermal fatigue, and more detrimental effects on inter-lamina shear and bending properties are observed [4].

2.1.4. Neutral environment

At the typical operational altitude for spacecrafts, including the space shuttle in LEO, the neutral atmospheric density is much less than that at sea level, which is about 10 orders of magnitude less. However, it is still large enough that, for instance, a 1-m^2 spacecraft orbiting at 8 km/s would suffer about 3×10^{22} collisions with ambient atoms every hour [1]. Undergoing such collisions, which cause the transfer of momentum to the

spacecraft, the spacecraft would experience a sizable amount of drag force during operation. This may result in its early entry to the atmosphere and require a large amount of fuel for thrusting. Moreover, the collisions cause the atomic neutral particles to chemically react with atomic particles from the surface of the spacecraft materials and consequently erode the surface.

In LEO, a major neutral constituent that is most hazardous toward polymers is AO. At an altitude of about 300 km, the densities of AO during maximum and minimum solar activities are approximately 8×10^9 and 2×10^9 atoms/cm³, respectively. If spacecraft orbits at a velocity of 8 km/s at the altitude, it would encounter with AO particles with kinetic energy of about 5 eV and the nominal AO flux ranging approximately from 10¹⁴ to 10¹⁵ atoms/cm² s [5]. Erosion of surface, loss in mass, degradation in mechanical, thermal and optical properties, and changes in chemical compositions of polymers and PMC's can be resulted through the collision with AO. For example, if graphite/epoxy composite materials are exposed to AO for 27.4 years at an altitude of 465 km, the erosion depth of the surface of the materials would be about 600 µm, which is equivalent with the thickness of 5 plies of the composite surface [2]. In general, such erosion by AO is associated with the degradation of material characteristics of polymers and PMC's. In addition, materials that resist the AO erosion effects are likely to create optical glow near material surface. The optical glow may hinder the visibility of the optical systems of any given space structure. It is thus recommended for spacecraft designer that in designing spacecraft using composite materials or spacecraft carrying sensitive optical payloads, proper selection of materials regarding the influences of AO be made beforehand.

2.1.5. Micrometeoroids and space debris

A huge number of small pieces of matter traveling at the speed ranged from 8 to 70 km/s exist in orbits around the Earth. If spacecraft orbiting the Earth at the typical speed for many orbits of 8 km/s is dashed against a small particle, it will cause a severe impact on the vehicle and even possibly bring a disastrous end of vehicle's useful life. For instance, under the same condition as above, the collision of the vehicle with a piece of matter of 0.3 cm in diameter would provide as much impact energy as a bowling ball moving at 97 km/s hitting the stationary vehicle [1].

Micrometeoroids and space debris are considered herein as the small pieces of matter that spacecraft collides with while orbiting the Earth. Micrometeoroids naturally exist in the solar system via breakups of comets, asteroids, etc. These space debris are of concern since they were, are still being, and will continue to be created by humans. Space debris are originated from launched objects; leftover pieces of satellite or booster rocket breakups such as battery, fuel, tank, and compressed gas tank; solid rocket fuel particles; operational debris such as lens cover, shroud, trash from manned spacecraft, etc.; spacecraft deterioration such as paint chips flaking; and other unknown objects. Currently, there are over 70,000 pieces of aspirin tablet sized debris in Earth orbits and the number historically has grown by 3–5% a year. It is known from a study [6] that a 10-m² spacecraft undergoes approximately 3100 collisions with micrometeoroids and space debris for 5.75 years.

Such collisions of space structures made of composite materials with micrometeoroids and man-made debris yield penetration through the composite structure, causing spallation damage in an extreme case. Even if the penetration does not occur, the collision can give rise to delamination of composite materials, which may also result in a fatal blow to the structure.

2.2. LEO space environment simulation facility

A LEO space environment facility capable of simulating the characteristics of LEO environment constituents, such as high vacuum, UV radiation, thermal cycling, and atomic oxygen atmosphere was designed and manufactured to study the characteristics and degradation mechanisms of composite materials after being exposed to LEO space environment. Fig. 1 presents a schematic of the simulation facility.

2.2.1. High vacuum

A main vacuum chamber was manufactured to simulate the ultra-high vacuum condition in LEO. Stainless steel that is resistant to deformation caused by thermal cycles, high vacuum, and outgassing was used as the main material of the chamber. The size of chamber is $500\% \times 400H$ (mm) so that relatively large sized material sample can be tested. The pumping system composed of rotary pump and diffusion pump for low and high vacuums, respectively, was used to produce the chamber pressure on the order of 10^{-6} Torr at a gas extraction rate of 10 L/s. Vacuum gauge system consists an ion senor measuring within a range of 1×10^{-3} – 10^{-10} Torr and a convectron sensor of 760– 1×10^{-3} Torr. A vacuum gauge controller was used to control the vacuum.

2.2.2. UV radiation

UV radiation of the wavelength of 100–200 nm is the primary electromagnetic radiation component in LEO environment [2]. The UV radiation source in the LEO simulation facility was thus manufactured using UV lamp carrying its wavelength of less than 200 nm. As shown in Fig. 1, the UV lamp was placed on exterior of the chamber to avoid excessive outgassing phenomenon on the source when placed inside the chamber. A quartz plate was placed between the UV radiation lamp

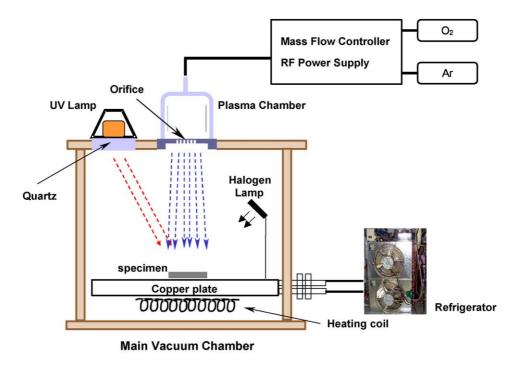


Fig. 1. Schematic diagram of LEO space environment simulation facility.

and the chamber so that the UV radiation could reach inside the chamber without loss of wavelength.

2.2.3. Thermal cycling

Thermal cycling from +150 °C to -150 °C takes place between the sun-facing and shadow-facing sides of the spacecraft while operating in LEO [7]. The sunfacing temperature was simulated using a halogen lamp, set inside the chamber, while the shadow facing temperature was simulated using a refrigerator circulating reusable coolant through a pipe placed inside a copper cooling plate. The copper plate on which the material samples were placed is inside the main chamber as shown in Fig. 1. A temperature sensor (T/C) was set on the copper plate to be aware of the temperature of specimen. The maximum and the minimum operating temperatures for the thermal cycling simulation are 100 °C and −70 °C, respectively. Temperature increases at a rate of approximately 5 °C/min and decreases approximately 3–4 °C/min; and a thermal cycle (+100 °C \rightarrow $-70 \,^{\circ}\text{C} \rightarrow +100 \,^{\circ}\text{C}$) takes about 64 min, as shown in Fig. 2.

2.2.4. Atomic oxygen

Atomic oxygen generation system equipped in LEO space environment simulation facility was manufactured to generate AO flux through weakly ionized remote oxygen plasma with a radio-frequency (RF) plasma source. The system is mainly operated through gas (O₂ and Ar) supply and 600 W, 13.56 MHz RF power supply, and pumping system to generate the vacuum condition in plasma chamber. Controlling gas flow rate of O₂ and

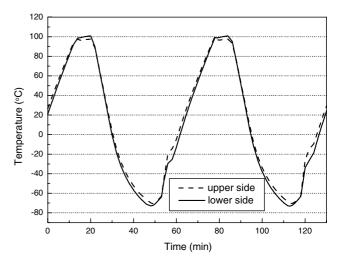


Fig. 2. Diagram of thermal cycling.

Ar (0–3.0 sccm), RF power (100–400 W), and orifice size in accordance with the plasma chamber pressure—that is a consequent factor for generating AO from molecular oxygen, the optimum AO flux was found herein. Table 1 shows configurations of five different orifices used.

It is of concern that the vacuum condition in the plasma chamber be on the order of 10^{-3} Torr in order to induce the highest quantity of density of AO. Moreover, the vacuum condition in the main chamber must be maintained within the level of LEO vacuum condition, regardless of neutral, plasma and electron fluxes, generated by RF plasma source, which enter the main chamber. Thus, it is crucial to select an adequate size of

Table 1 Orifice configurations

Orifice	Number of holes	Hole diameter (mm)	Total area of holes (mm ²)
A	61	2	191.64
В	154	1	120.95
C	119	1	93.46
D	69	1	54.19
E	69	1	26.55

orifice. Fig. 3 shows the plots of vacuum condition in the main vacuum chambers and the plasma chamber, vs. orifice sizes, at a total gas flow rate of 5.0 sccm. It is recommended that the orifice size used for this system must be as large as possible, because the conductance of any species (AO neutrals) from plasma chamber to main chamber increases as the orifice size increases. As shown in Fig. 3, the five different orifices without exception satisfied the vacuum condition for LEO space environment since the highest pressure of the main chamber was less than 5.0×10^{-5} Torr at the total gas flow rate. However, the orifice A with 2-mm diameter and 61 holes was chosen to be used in this system, because of its largest size.

Plasma discharge simulation, which was coded based on the rate constants for a restricted set of two body reactions of interest in modeling low-pressure oxygen discharges [8], was used to determine the maximum AO flux through encoding a number of different plasma parameters such as O2 and Ar gas flow rates, RF power (100-400 W) and plasma chamber pressure. Regarding the vacuum condition of the main chamber, the maximum total gas flow rate was determined at 5.0 sccm and the orifice A with 2-mm diameter and 61 holes was selected for the simulation. The plasma discharge simulation was subsequently performed to estimate the optimum gas composition and RF power, which induces maximum density of AO for the oxygen plasma. The estimated optimum gas composition of oxygen and argon (O2:Ar) and RF power were 0.2:0.8 and 200 W, respectively, and the maximum AO density was approx-

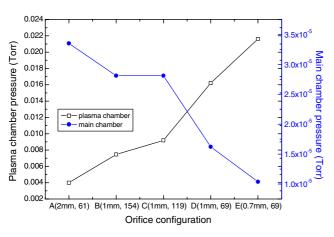


Fig. 3. Main chamber and plasma chamber pressures vs. orifice size.

imately $9.77 \times 10^{13} \, \mathrm{cm}^{-3}$. Fig. 4 shows densities of neutral species in plasma as a function of RF power at a total gas flow rate of 5.0 sccm with the optimum gas composition. The maximum AO flux was then calculated through the equation as follows:

$$\Gamma_{\rm N} = \frac{1}{4} N v_{\rm N}^{\rm TH}$$

where $\Gamma_{\rm N}$ is flux of neutral species, N is density of neutral species, and $v_{\rm N}^{\rm TH}$ is thermal velocity of neutral species. The expression for $v_{\rm N}^{\rm TH}$ takes the formula

$$v_{\rm N}^{\rm TH} = \sqrt{8T_{\rm N}/\pi M}$$

where $T_{\rm N}$ is temperature of neutral species, and M is mass of neutral species [8]. The estimated kinetic energy of atomic oxygen neutrals in the oxygen plasma was approximately 0.04 eV. The calculated maximum AO flux was approximately 6.05×10^{17} atoms/cm² s.

AO flux encountered upon the specimen in the main chamber, which was placed 341-mm below the orifice (Fig. 1), was determined by estimating the divergence angle of the AO beam, shown in Fig. 5. The estimation of the divergence angle was conducted through measuring the diameter of AO beam exposed to a composite sample as a function of distance from the orifice. Fig. 6 shows the trace of AO beam exposure on the sample placed 125 mm below the orifice for 24 h. Obtaining the diameters of the trace and orifice and the distance between them, which were 55 mm, 35 mm and 91 mm, respectively, the divergence angle of AO beam was calculated to be approximately 6.27°.

AO flux encountered upon the specimen (Fig. 1) in this system, which was placed 341-mm below the orifice, was determined to be approximately 6.14×10^{16} atoms/cm² s and plasma energy was about 0.04 eV. Regarding the nominal AO flux in LEO (250 km, the flux $\Phi \approx 3 \times 10^{14}$ atoms/cm² s), it is said that the LEO space environment simulation facility offers a suitable accelerated simulation testing of AO environment in LEO.

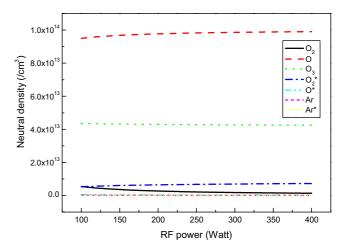


Fig. 4. Density of neutral species vs. RF power.

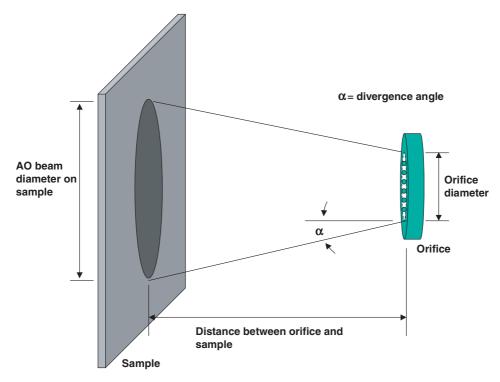


Fig. 5. Schematic diagram of divergence angle of AO beam, α .

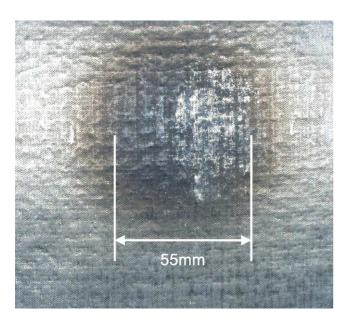


Fig. 6. Trace of AO beam on composite sample.

3. LEO space environment characteristics test

Mass losses, along with mechanical property changes of graphite/epoxy composite materials after the synergistic LEO space environment exposure and AO exposure, were investigated herein to evaluate the space environmental characteristics of the composite materials and performance of the composite structures. The aging period of simulated LEO environment exposure was

 $5 \, \text{h}$ and $45 \, \text{min}$. The AO fluence of about $1.27 \times 10^{21} \, \text{atoms/cm}^2$ and 4 thermal cycles took place over the aging period for the LEO synergistic exposure.

3.1. Mass loss test

Change of mass occurs on composite space structures if spacecrafts operate for long period of time in hazard-ous space environment, in which composite material deteriorate by the space environmental effects. Its mass is then easily lost through outgassing. Thus the outgassing deforms the structure of spacecraft, causing contamination and failure of dimensional stability.

The mass loss test measures the total mass loss (TML) of the degraded material by the LEO space environment effects, caused by outgassing under vacuum condition. TML defines the total mass of material, outgassed from specimen during a particular time period at a particular pressure. TML is calculated with the initial specimen mass and the final specimen mass, measured before and after the test, respectively, using a super precise scale (METTER AF260 DeltaRange, Inc.) with an accuracy of 10⁻⁴ g, and expressed as percentage (%) of the initial mass. TML is calculated with a simple equation

$$\left(\frac{S_{\rm I} - S_{\rm F}}{S_{\rm I}}\right) \times 100 = \% \text{TML}$$

where $S_{\rm I}$ is the initial specimen mass and $S_{\rm F}$ is the final specimen mass. Composite specimens for mass loss test

were fabricated using HFG-CU-125NS graphite/epoxy prepreg tape, the thickness of which is 0.125 mm. The dimension of the specimens manufactured was $7 \times 7 \times 1$ mm.

3.2. Tensile properties test

Space environmental effects on tensile characteristics of composite materials must be studied for space application of composite materials because it is directly associated with the damage of composite structures in space. Thus, the changes of tensile properties of composite materials after a certain period of exposure to AO and synergistic space environment were investigated.

ASTM D3039 M-95 was used as a tension test method to measure the tensile strength and stiffness of the specimen. The dimension of the tensile test specimens was determined at $140 \times 12.7 \times 1$ mm, calculated according to the tensile specimen geometry requirements noted in ASTM D3039 M-95. This specimens were then manufactured with 0° unidirectional fiber orientation using the HFG-CU-125NS graphite/epoxy prepreg tape.

4. Results and discussion

4.1. Mass loss

The TML's of graphite/epoxy specimens exposed to AO and LEO synergistic space environment over the aging period are shown in Fig. 7. The TML's after AO exposure and synergistic LEO environment exposure were approximately 0.75% and 1.51%, respectively. The mass erosion of the specimens under synergistic LEO environment effects was much severe than that under AO effects. In a study by Tennyson [9], UV radiation significantly enhances the mass erosion rate by the AO

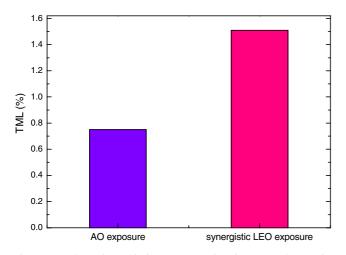


Fig. 7. Mass loss of graphite/epoxy composite after AO and synergistic LEO environment exposures.

exposure. It is apparent that the synergistic effects of LEO space environment, consisting AO exposure and UV radiation, increases the mass loss rate of the graphite/epoxy composite, as compared to only AO effects. Moreover, thermal cycling added to the synergistic LEO effects probably activated the mechanism of mass loss of the graphite/epoxy composite.

4.2. Tensile properties

The tensile strengths of graphite/epoxy composites unexposed and exposed to AO and synergistic LEO environment were 2906.4 MPa, 2811.3 MPa and 2710.0 MPa, respectively, as shown in Fig. 8. The tensile strength of the composite specimen was degraded 3.27% and 6.76% after being exposed to AO and the synergistic LEO environment, respectively. Fig. 9 shows the tensile stiffness of the composite specimens. The tensile stiffness of graphite/epoxy composites unexposed and exposed to AO and synergistic LEO environment were 142.9 GPa, 137.2 GPa and 130.9 GPa, respectively, and 3.99% and 8.4% of reductions in tensile stiffness were observed.

Regarding the changes of tensile properties, it can be said that the tensile properties of the graphite/epoxy composites are significantly influenced by the synergistic effects of LEO space environment constituents (AO, UV radiation and thermal cycling). The synergistic LEO space environment effects, which induce merged damages on the composite structures such as microcracking by thermal cycling [10] and mass loss by AO and UV radiation, considerably deteriorated the tensile characteristics of the graphite/epoxy composites more severely than by only the AO effects.

4.3. Surface morphology

Surface morphologies of the unexposed, only AO exposed and synergistic LEO environment exposed speci-

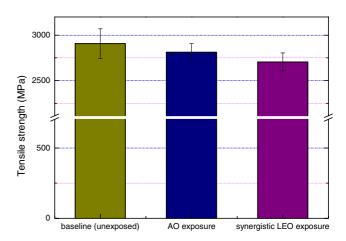


Fig. 8. Tensile strength of graphite/epoxy composites unexposed and exposed to AO and synergistic LEO environment.

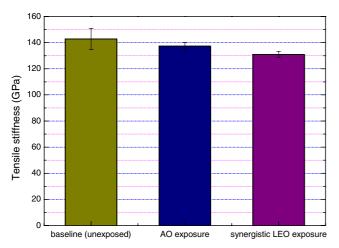
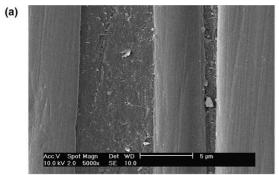
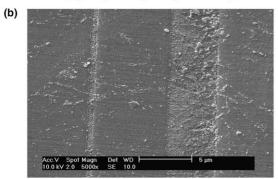


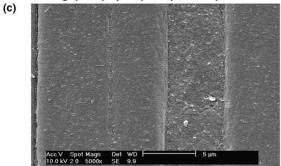
Fig. 9. Tensile stiffness of graphite/epoxy composites unexposed and exposed to AO and synergistic LEO environment.



graphite/epoxy composite specimen unexposed



graphite/epoxy composite specimen exposed to AO



graphite/epoxy composite specimen exposed to the synergistic LEO environment

Fig. 10. SEM surface morphology of graphite/epoxy specimens unexposed/exposed to the LEO space environment effects (5000×).

mens were taken by SEM (5000×) in order to observe the changes of surface appearance of the specimen caused by LEO environment effects, as shown in Fig. 10. It can be seen from Fig. 10 that the surfaces of the both specimens exposed to AO and the synergistic LEO environment are somewhat eroded. In comparison, composite specimen exposed to the synergistic LEO environment shows a number of microcracks on fibers and matrix in addition to AO erosion. Thus, the severest effects on mass loss and tensile properties of the graphite/epoxy composites is shown to be caused by the synergistic LEO space environment.

It is apparent that the synergistic LEO space environment effects on the composite materials are very destructive. Among all the constituents in LEO space environment, AO exposure is the major constituent to erode the mass of composites; however, it's mass erosion effects become severer when combined with UV radiation. And, the tensile (material) characteristics of the composites are significantly degraded by the synergistic LEO space environment effects.

5. Conclusion

In this paper, in order to investigate the LEO space environment characteristics of composite materials and to acquire the reliable understanding of the LEO space environment effects on composite materials, a LEO space environment simulation facility capable of simulating major LEO conditions, characteristics of which are high vacuum (10^{-5} – 10^{-6} Torr), UV radiation (<200 nm wavelength), thermal cycling (100° C to -70° C to -100° C), and atomic oxygen atmosphere (kinetic energy of ~ 0.04 eV and nominal flux of $\sim 10^{16}$ atoms/cm² s), have been designed and manufactured. Furnishing the LEO space environment simulation facility, study on the LEO space environment characteristics of composite materials was successively carried out.

Changes in mass, stiffness and tensile strength and surface morphology of graphite/epoxy composite specimens after being exposed to the simulated LEO space environment have been acquired. It is observed from the changes by AO and synergistic LEO exposures that AO erodes the surface of graphite/epoxy composites, causing mass loss and degradation in tensile properties under vacuum condition, while the synergistic LEO exposure induce harsher effects. For example, when UV radiation and thermal cycling are combined with AO exposure, more excessive surface (mass) erosion, as well as microcracks, takes place on the composite specimen. It can be concluded that the LEO space environment consists of hazardous constituents to composite materials and its synergistic effects induce significant changes in mass, tensile properties, and surface morphology, causing considerable damage to the composite structures.

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