The effects of space atomic oxygen erosion on epoxy and silicone adhesives in LEO spacecraft

Li Tao, Jiang Lixiang, Feng Weiquan, Liu Xiangpeng
(Beijing Institute of Spacecraft Environment Engineering, Beijing 100094, China)

Abstract: Atomic oxygen (AO) is prevalent in Low Earth Orbit (LEO). Ground-based testing on AO exposure was performed to investigate the effects of atomic oxygen on materials to be used in LEO spacecraft. Two types of adhesive materials, E-32 epoxy and GS414 silicone, were tested in the atomic oxygen testing facility at Beijing Institute of Spacecraft Environment Engineering (BISEE). The equivalent atomic oxygen fluence in test was approximately 1.4×10^{21} atom/cm^2 as determined based on two years' exposure on LEO. Significant mass losses of the epoxy adhesive samples were observed after the exposure to atomic oxygen, but relatively small mass changes were found in the silicone samples. The erosion yields of epoxy samples range from 3.2×10^{-24} cm^3/atom to 3.8×10^{-24} cm^3/atom. Bleaching by atomic oxygen was found in the epoxy samples. The surface on the silicone sample was glossed after AO exposure. The external appearance of both kinds of materials was analyzed using Scanning Electron Microscope (SEM) and X-ray Photoelectron Spectroscopy (XPS). A comparison between unexposed and exposed samples shows noticeable changes on the surface of materials.

Key words: atomic oxygen; epoxy resin; silicone; erosion effects
CLC number: O631.2; V416.5 **Document code: A** **Article ID: 1673-1379(2009)03-0222-03**
DOI: 10.3969/j.issn.1673-1379.2009.03.005

1 Introduction

The low Earth orbit (LEO), 200~700 km above the Earth surface, sees a harsh space environment with hazards such as atomic oxygen, ultraviolet (UV) radiation, ionizing radiation (electrons, protons), high vacuum, micrometeoroids and debris, as well as severe temperature cycling, which may cause considerable damages to spacecraft materials. Atomic oxygen is predominant among those hazards[1,2]. It is formed by solar ultraviolet radiation, which dissociates oxygen molecules (O_2) into free atomic oxygen in the outer ionosphere at an altitude higher than 100 km.

Various materials in various forms are used in space exploration, including organic materials, metallic and nonmetallic materials, optical coatings, and thermal control coatings. Those materials will be exposed to space atomic oxygen environment if used for LEO space flight mission, and some of them may suffer from mass loss, function degradation, which may lead to reduced reliability of spacecraft[3]. Ground-based test is usually carried out to evaluate materials selected for space missions due to the consideration of saving time and cost.

Two types of adhesive materials were selected in our tests to study their responses to atomic oxygen.

2 Test facility

The samples were tested in the AO facility of BISEE. Figure 1 gives a schematic view of the neutral atomic oxygen source, with a plasma chamber containing a electron cyclotron resonance (ECR) plasma source, a neutralizing plate, a pumping system and other auxiliary instruments. The plasma source generates dense low-temperature plasma, which flows in the magnetic field to ionize O_2 molecules; the oxygen ions collide with the Mo plate (with a negative potential) to form an atomic oxygen beam. The sample holder is placed downstream in the flow of atomic oxygen.

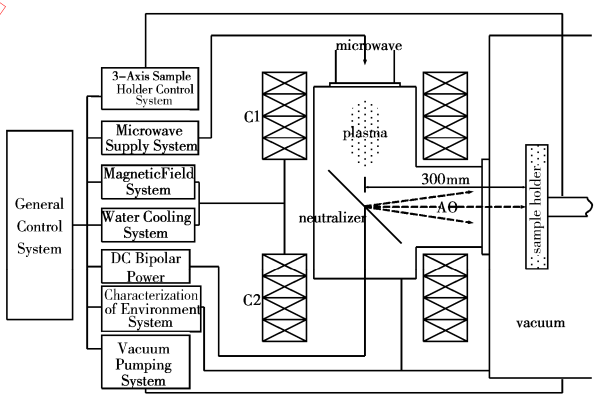


Fig. 1 Schematic view of AO simulation facility

The facility can produce an AO beam of a flux of approximately 4×10^{15}atom/(cm^2·s) with energy of 3~10 eV 300 mm away from the center of the neutralizer. The beam is nearly uniform within an area of ϕ 150 mm at the position, thus many test samples and witness samples can be placed on the sample holder and with almost the same amount of AO exposure at every instant. Kapton HN film is used to monitor AO flux and fluence.

The ECR source is expected to produce vacuum ultraviolet (VUV) radiation with a center wavelength of 130.4 nm due to

收稿日期：2008-10-14
基金项目：国家 863 项目支持（编号：2006AA04Z414）
作者简介：李 涛（1981-），男，硕士学位，工程师，主要从事空间原子氧环境试验技术研究。E-mail：litaohit@163.com。

O_2 dissociation and ionization during plasma generation. VUV radiation may also have effects on polymers, so CsTe photodiodes with MgF_2 window were used to detect the radiation in the range from 116 nm to 254 nm.

3 Atomic oxygen test

3.1 Test materials

(1) E-32 Epoxy

Epoxy is a very important class of thermosetting resin used in engineering due to its outstanding strength of adhesion with most kinds of surfaces, high mechanical strength, and chemical resistance. It is used as adhesives, coatings and resin matrices for advanced composites[4]. E-32 epoxy is composed of epoxy oligomer, a kind of propyl curing agent, and acetone as the thinner. This kind of epoxy is widely chosen as an adhesive on the surface of spacecraft.

(2) GS414 Silicone

Room temperature vulcanization silicone rubber can be vulcanized at room temperature without small molecule emission, heat generation or shrinkage. Silicone rubber enjoys many excellent properties, such as short setting time, fine dimensional stability, low compression deformation, and the vulcanization rate can be easily controlled by temperature. So it is widely applied in electronics, electric appliances, aerospace vehicles, and outer space missions. GS414 Silicone is a silicone adhesive that will be used on spacecraft in future missions[5].

GS414 samples are about 1.0 mm thick and 10 mm in diameter. They were all cleaned by pure ethanol, and then placed in a clean vacuum chamber (with vacuum degree $\leqslant$20 Pa) for 24 h prior to atomic oxygen experiment.

3.2 Simulated environment

The tests were designed to determine atomic oxygen reactivity of the above-mentioned two types of adhesive materials, and subsequently, to forecast the effects of two years' AO exposure in LEO on materials properties.

a. Effective flux: 2×10^{15} atom/(cm^2·s)

The beam flux at the sample position was indirectly obtained by measuring the mass loss of Kapton HN, whose in-space erosion yield is 3×10^{-24} cm^3/atom according to the flight experiment results.

b. Effective fluence: 1.4×10^{21} atom/cm^2

The effective fluence of atomic oxygen in the test was calculated using a software named AOFF-MCDD, which evaluates the AO environment on the surface of LEO spacecraft based on their altitude, life period and other mission parameters[6].

The fluence is also determined by using Kapton HN.

c. Work pressure: 2.0×10^{-2} Pa

d. Sample temperature: below 40℃

e. Radiation interference: negligible

In the pre-testing, CsTe photodiodes and Faraday cup were used to determine the level of background VUV and ions. Results show that no VUV radiation and very little uncontrolled ions were found at the sample position in the simulated atomic oxygen environment.

4 Analysis of results

4.1 Visual inspection

Epoxy samples are dark, not very reflective, with white powders clearly visible on the surface.

The surface of silicone samples is yellowish, raised, and appears glossed after AO exposure.

4.2 Mass loss

Table 1 shows the mass loss of the samples.

Table 1 Mass loss of the test samples

Effective AO fluence: 1.4×10^{21} atom/cm^2

Material	E-32 epoxy		GS414 silicone	
ID No.	1#	2#	3#	4#
Total Mass Loss/g	0.007 78	0.009 03	0.000 03	-0.000 06

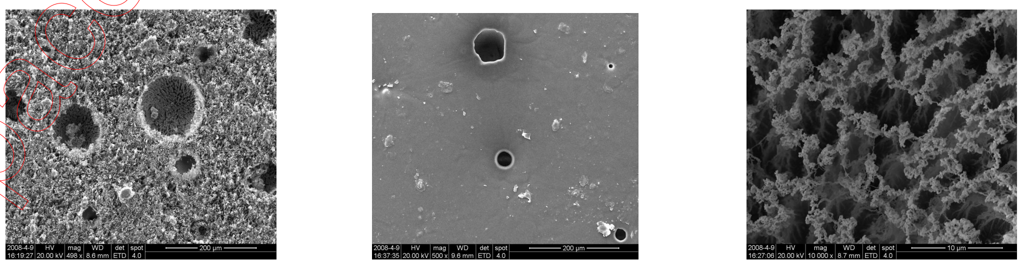
With the exposure to atomic oxygen, for epoxy, the mass loss of samples is linearly related with the effective fluence of atomic oxygen according to the test results; for silicone, however, the mass of samples changes very little.

4.3 Oxygen erosion

The E-32 epoxy erosion yield is in a range from 3.2×10^{-24} cm^3/atom to 3.8×10^{-24} cm^3/atom based on the above mass loss results; but for GS414 silicone, the erosion yield is significantly smaller.

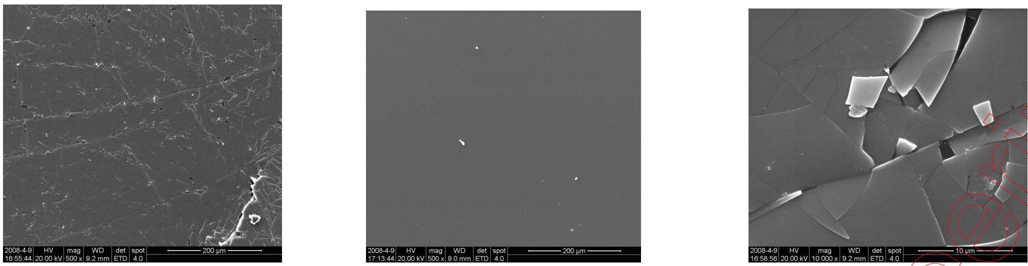
4.4 SEM analysis

Views of an epoxy sample and a silicone sample are shown in Fig. 2 and Fig. 3, respectively, using FEI Nova 400. After atomic oxygen exposure, the surface of the epoxy sample is roughened, and a three-dimensional network structure is seen in the erosion area. For silicone, visible cracks are observed on the sample surface, with many glasslike chippings around surface cracks. Cracking stress is considered to be developed on the surface of the material after the atomic oxygen exposure.



(a) Exposed (500X) (b) Unexposed (500X) (c) Exposed (10 000X)

Fig. 2 Views of exposed and unexposed epoxy samples using FEI Nova 400



(a) Exposed (500X) (b) Unexposed (500X) (c) Exposed (10 000X)

Fig. 3 Views of exposed and unexposed silicone samples using FEI Nova 400

4.5 XPS analysis

XPS was used to analyze the changes in the elemental composition of the materials tested. Table 2 shows the main elemental data of two kinds of materials before and after the atomic oxygen exposure. The results indicate that C and O are the main elements, and there are a small quantity of N, S, and Si in the epoxy specimen. After atomic oxygen test, the contents of C, N and Si on the surface of E-32 epoxy fall off, but that of O increases. For GS414 silicone, the content of C increases, but those of O and Si fall off. Mo is also found on the surface of samples after the exposure, but in a very small quantity.

Table 2 Main relative elemental compositions of the test samples

Unit:%

Element	E-32 epoxy		GS414 silicone	
	exposed	unexposed	exposed	unexposed
C 1s	59.23	71.25	48.86	35.41
N 1s	0.00	5.95	0.00	0.00
O 1s	31.26	21.12	28.19	38.56
Si 2p	0.00	1.21	20.39	26.03

5 Conclusions

Two kinds of adhesive materials were tested to evaluate the effects of AO exposure of a fluence equivalent to two years' LEO on their properties. The results indicate that:

1) For E-32 epoxy adhesive, the erosion yield ranges between 3.2×10^{-24} cm^3/atom and 3.8×10^{-24} cm^3/atom. Two years' AO exposure can result in thickness loss, white powders appearing on the surface, and chemical and physical changes in the exposed area.

2) GS414 silicone has some resistance against atomic oxygen erosion. The mass loss is very little as shown by a comparison between the exposed and unexposed samples, but great changes may be observed on the surface of the material after atomic oxygen attack, including shiny products, many visible cracks and chemical changes.

This paper may provide designers of spacecraft some food for thought in determining whether, where and how to use these two kinds of materials in the future mission.

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空间原子氧对低地球轨道航天器用粘结剂的腐蚀效应影响

李 涛，姜利祥，冯伟泉，刘向鹏

（北京卫星环境工程研究所，北京 100094）

摘要：原子氧是低地球轨道（LEO）一种重要的空间环境因素，地面模拟试验是用于开展 LEO 航天器备选材料原子氧环境评价的主要手段。利用北京卫星环境研究所的原子氧设备，对牌号为 E-32 的环氧树脂和牌号为 GS414 的硅橡胶两种粘结剂进行了评价试验。依据轨道参数与航天器两年的设计寿命，选定试验中原子氧的等效积分通量为 1.4×10^{21} atom/cm^2。试验结果表明，环氧树脂样品出现了较大的质量损失，而硅橡胶样品的质量损失则相对较小。根据质量损失计算环氧树脂样品的原子氧反应率处于 3.2×10^{-24} ~ 3.8×10^{-24} cm^3/atom 之间。试验后的环氧树脂样品表面颜色变浅，硅橡胶样品的表面则呈现了玻璃化。对两种材料都进行了表面形貌分析，试验前后测试结果的对比分析表明原子氧的氧化作用使材料表面发生了较大的变化。

关键词：原子氧；环氧树脂；硅橡胶；腐蚀效应