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Study on Sequent Test Effects of Atomic Oxygen/thermal Cycling

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Abstract. Two kinds of unidirectional laminates made from carbon/epoxy composite named M40/DFA-1 & M40/DFA-N3(nano TiO₂ contained) are selected to study the different effects between different test sequences of atomic oxygen/thermal cycling. Based on the ground simulating test of atomic oxygen and thermal cycling, several kinds of tests and analysis are conducted, such as surface morphology observation, mass loss test, inter-laminar shear strength test, and X-ray photoelectron analysis of surface components. After the exposure of atomic oxygen and thermal cycling, surface erosions is observed on both of the materials, in addition, mechanical properties are both changed. It must be highlight that, under the condition of 1.5×10^{20} atoms/cm² AO and 200 thermal cycles within the range of -150°C~+150°C, different sequences lead to different results, either the extend or the tendency. In which, samples under AO+TC sequence exhibited relatively higher mass loss and erosion rate as well as more severe mechanical performance degradation.

1. Introduction

In the Low Earth Orbit(LEO) environment where some of the spacecraft would be located, surfaces will be exposed to potentially damaging environment such as atomic oxygen, ultraviolet radiation, charged particles radiation, thermal cycling, debris and so on[1,2]. Synergistic effects may exist between different space environment factors[3]. Therefore, under the circumstances of ground-based facilities, basing on the acknowledgement of single factor effects, it is of great importance to discover the principles of performance degradation, and to figure out what's going on.

Atomic oxygen and thermal cycling are referred as the most principal factors among LEO environment[4]. In which, atomic oxygen constitutes the primary components of neutral atmosphere, furthermore, it will harshly impact on the surface of spacecraft at the relative speed of 8km/s, and leads to severe erosion of the surface material[5-8]. Meanwhile, thermal cycling between extreme temperatures is another inevitable environment when spacecraft orbits through the shadow area in and out over and over again.

On the basis of applications in the engineering[9-12], two kinds of unidirectional laminates made from carbon/epoxy composite named M40/DFA-1 & M40/DFA-N3(nano TiO₂ contained) are selected in this work, to do the research on the different effects result from different test sequence, focusing on the environment of atomic oxygen and thermal cycling.

2. Experimental Procedure

2.1. Test Conditions and Materials



Atomic oxygen impact fluence is set to be 1.5×10^{20} atoms/cm². To avoid the effect of micro-cracks on the mechanical properties of testing materials during thermal cycling, temperature range is chosen as -150°C~+150°C according to other references, with 200 cycles in all.

Epoxy resin composites enhanced by carbon fibers M40/DFA-1, and 3% nano TiO₂ contained epoxy ester composites enhanced by carbon fibers M40/DFA-N3 are exposed to the test environments as mentioned respectively.

2.2. Instruments and Methods

The atomic oxygen tests and thermal cycling tests are conducted respectively on the facilities at Beijing Institute of Space Environment Engineering.

The sample's mass loss is measured by German Miro Sartorius precision electronics balance, with the accuracy of 10^{-5} . Mass data before and after environment test have been recorded respectively, and the mass loss rates are calculated as following:

$$M_{\text{loss}} = [(M_0 - M_t) / M_0] \times 100\% \quad (1)$$

Where, M_0 is the original mass of specimen, g; M_t is the mass of specimen after environment test, g.

The inter-laminar shear strength is tested based on the GB/T 30969-2014 standard by applying the three-point short beam method, at the loading rate of 1.3 mm/min, calculated as following:

$$\text{ILSS} = \frac{3P_{\text{max}}}{4bd} \quad (2)$$

Where, P_{max} is the maximum of the breaking load, N; b is the width of the specimen, mm; d is the thickness of the specimen, mm.

The optical surface morphology is observed by Olympus GX-51 optical microscope; the micro morphology is observed by Scanning Election Microscope (SEM) of the type MX2600FE; the surface element analysis is conducted on the basis of XPS(X-ray Photoelectron Spectroscopy) by VG ESCALAB MK II.

3. Results and Analysis

3.1. Optical Surface Morphology

Generally speaking, most of the polymer-matrixed composites are relatively vulnerable to the atomic oxygen(AO) and thermal cycling(TC). Specimen of both materials are observed before and after AO&TC sequent tests, as shown in Fig.1 and Fig.2 In the view of optical microscope, both surfaces have turned dark, but phenomenon can't be distinguished obviously between different test sequence.

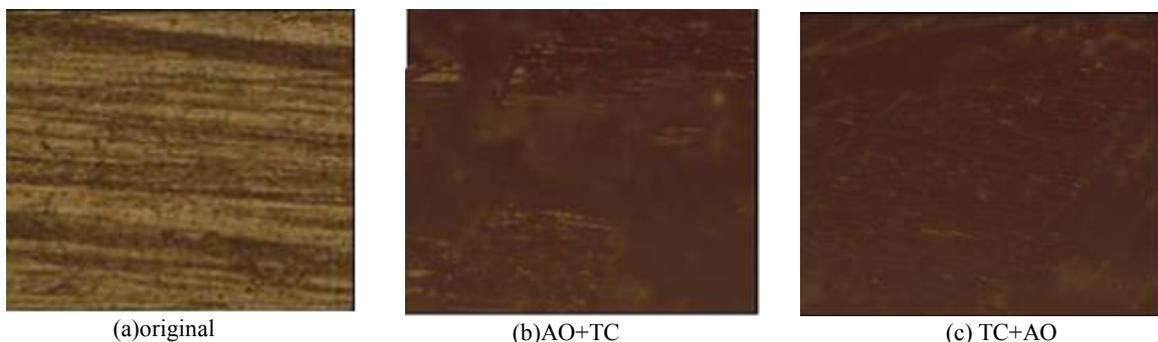
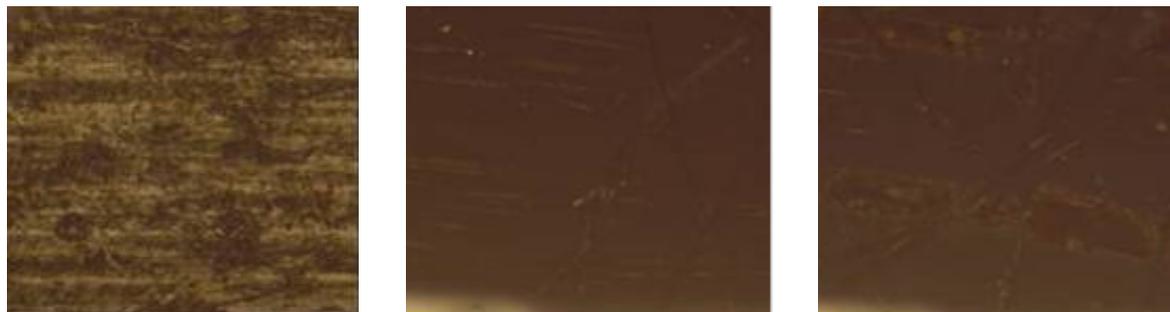


Figure 1. Surface of the M40/DFA-1 specimen before/after AO&TC sequent tests



(a)original (b)AO+TC (c)TC+AO
Figure 2. Surface of the M40/DFA-N3 specimen before/after AO&TC sequent tests

3.2. Mass Loss Rate

Masses of the specimens made from the both materials have been weighed before and after the bidirectional sequent tests, in addition, mass loss has been calculated, as well as the mass loss rate. The results are shown in the Table 1.

Table 1. Mass loss rate of the specimen under AO/TC sequence test

Types of the Materials	Mass Loss Rate	
	AO+TC	TC+AO
M40/DFA-1	0.425%	0.193%
M40/DFA-N3	0.456%	0.199%

As the data reveals, for both materials, the mass loss rates after AO+TC test are obviously higher than that after TC+AO test. To discuss the reason beneath the phenomenon, it may be possible that atomic oxygen brought the roughness to the materials surfaces, which increased the specific surface area, thus, the composites contact area interacted with C,N,H in the air has raised, and released more volatile gases like CO, CO₂ and H₂O, therefore, raised the mass loss rate.

On the other hand, mass loss after TC+AO sequent test are obviously lower, the primary reason may be the post curing process of the polymers undergoing thermal cycling exposure. The procedure of post curing could enhance the crosslinking density of resin matrix, which tightens the combination between atoms, as a result, strengthens the materials' resistance to atomic oxygen.

The data indicates that, as far as atomic oxygen and thermal cycling is concerned, different test sequences lead to damage with distinction. Given the conditions in this work, the sequence of AO+TC induced higher mass loss than that from TC+AO sequence.

3.3. Inter-laminar Shear Strength

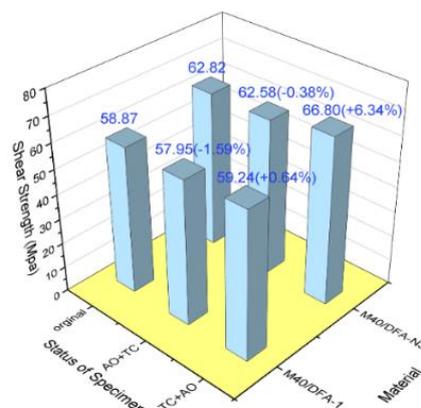


Figure 3. ILSS of specimens before/after AO&TC sequent test

As illustrated in Fig.3, is the inter-laminar shear strengths of specimens before and after the sequent tests. The data shows that the inter-laminar strengths of both materials are declined after AO+TC

sequent test, of which M40/DFA-1 dropped from 58.87MPa to 57.95MPa, about 1.59% reduction, while M40/DFA-N3 dropped from 62.82MPa to 62.58MPa, about 0.38% reduction. On the other hand, inter-laminar strengths of both materials are enhanced after TC+AO sequent test, of which M40/DFA-1 raised to 59.24MPa at the rate of 0.64%, while M40/DFA-N3 raised to 66.80MPa, at the rate of 6.34%.

Thus, given the conditions of atomic oxygen and thermal cycling test, different test sequence leads to different effects in mechanical property.

Basically, according to the related researches, atomic oxygen leads to the declination of the mechanical performance, whereas, thermal cycling usually strengthens it (in the circumstances without micro-cracks occur). From that point of view, we deduce that, in the AO+TC sequent test, atomic oxygen did more severe damage to the mechanical property than that of thermal cycling, which resulted in mechanical reduction; while in the TC+AO sequent test, exposure in thermal cycling brought performance promotion to the mechanical performance directly in the way of raising the crosslinking density, besides, this procedure itself could also reduce the erosion from atomic oxygen.

3.4. Surface Micro Morphology

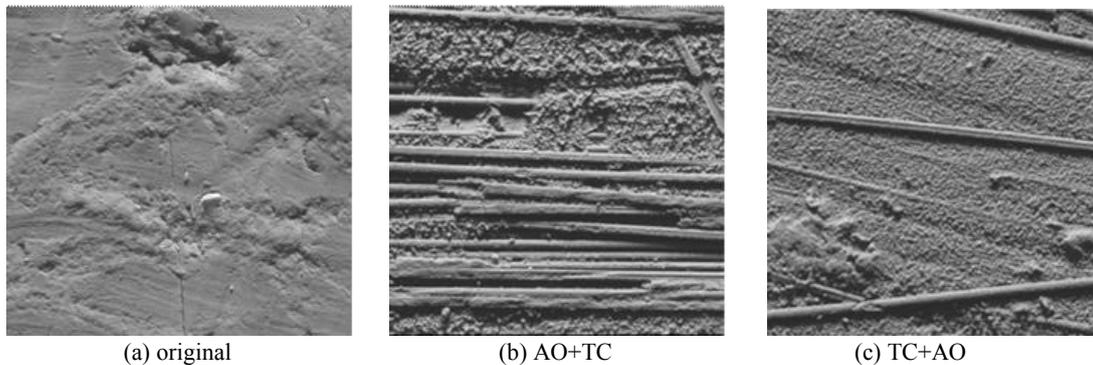


Figure 4. Surface morphology of M40/DFA-1 before/after AO&TC sequent tests (SEM)

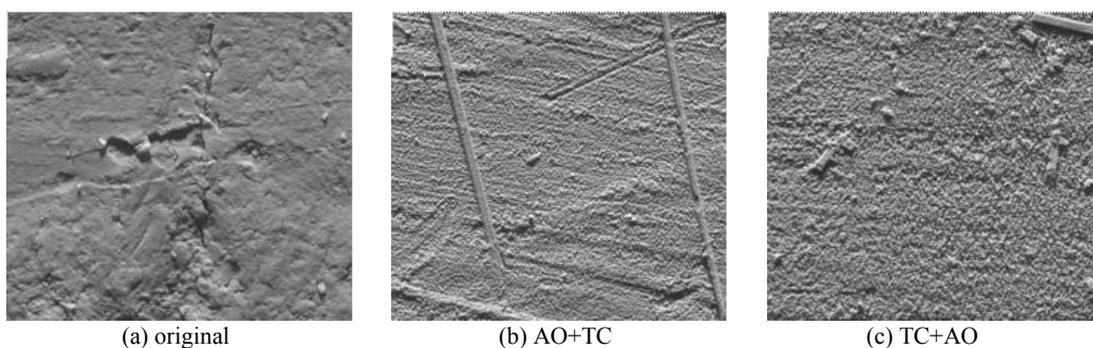


Figure 5. Surface morphology of M40/DFA-N3 before/after AO&TC sequent tests (SEM)

As illustrated in Fig.4 and Fig.5, surface micro morphology was observed before and after bidirectional sequent tests by the Scanning Election Microscope (SEM).

It is obvious that, the resin was denudated seriously on the surface of M40/DFA-1, leaving plenty of near-face fibers exposed outwardly, with several carbon fibers ruptured; otherwise, under the sequent test of TC+AO, resin on the surface is also eroded, leads to “carpet” morphology, but, relatively unobvious.

Similar phenomenon appeared on the surface of M40/ DFA-N3.It is visible that single fibers exposed on the surface after AO+TC sequent test, while the erosion is less severe after TC+AO sequence, with no fibers on the surface exposed.

3.5. Analysis of Surface Components

Detailed C1s peak spectrums XPS analysis of M40/DFA-1 before and after sequent tests are illustrated in Fig.6 as follows. The peaks of element of C in organic molecules are usually located at 285 eV(C-C bond), 286.5 eV(C-O-C bond), 287.6 eV(CHO bond), and 288.6 eV(COOH bond)[13]. As is shown in the graphs, C1s spectrums of both materials changed after sequent tests, with distinguished appearances: under the sequence of AO+TC, the specific area of the peak at 285 eV which refers to the C-C bond, shrank sharply, while that of TC+AO seemed little; which refers to the C-O-C bond expanded after AO+TC test, whereas that of TC+AO test diminished; besides, the specific area of the peak at 289 eV referring to the C=O bond heightened slightly after both sequences. Data as mentioned are laid in Table 2.

The result indicates that thermal cycling retained more C-C from de-bonding by atomic oxygen, weakening the effects of atomic oxygen. Basically, if an oxygen atomic breaks in the C-C bond to form a C-O-C bond, it would then reduce the crosslinking density, therefore, in the contrast between AO+TC and TC+AO, the latter maintained a relatively higher crosslinking density, furtherly, the mechanical property. That is matching with the inter-laminar strength measurement.

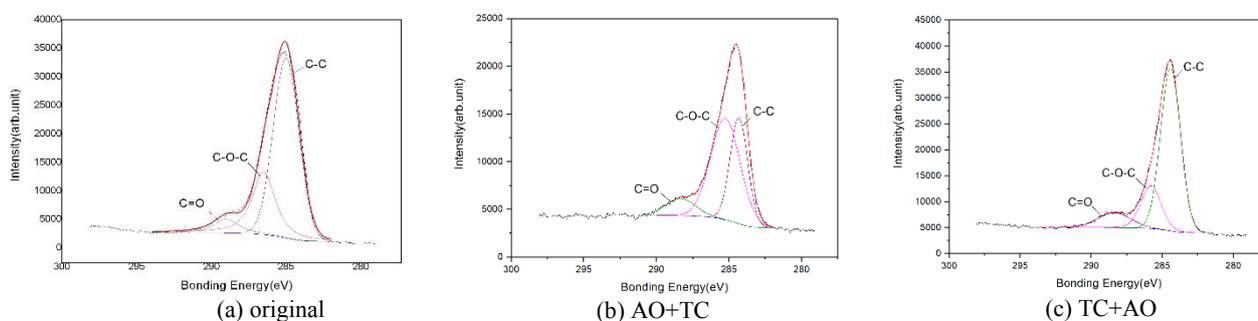


Figure 6. C1s spectrum of M40/DFA-1 specimen surface before/after AO&TC sequent tests

Table 2. XPS analysis of M40/DFA-1 before/after AO&TC sequent tests

Functional Groups	Bonding Energy/eV	Relative Percentage of Specific Area/ % original/(AO+TC)/(TC+AO)
C-C	285.000	70.20/34.83/69.8
C-O-C	286.500	27.82/55.80/19.58
C=O	289.000	6.66/9.37/10.62

Detailed C1s peak spectrums XPS analysis of M40/DFA-N3 before and after sequent tests are illustrated in Fig.7 as follows. The result is basically consistence with that of M40/DFA-1: more C-C bonds are retained in TC+AO sequent test than in AO+TC (specific data are laid in Table 3) , which means a higher crosslinking density. So the mechanical property performed higher, which is consistent with measurement.

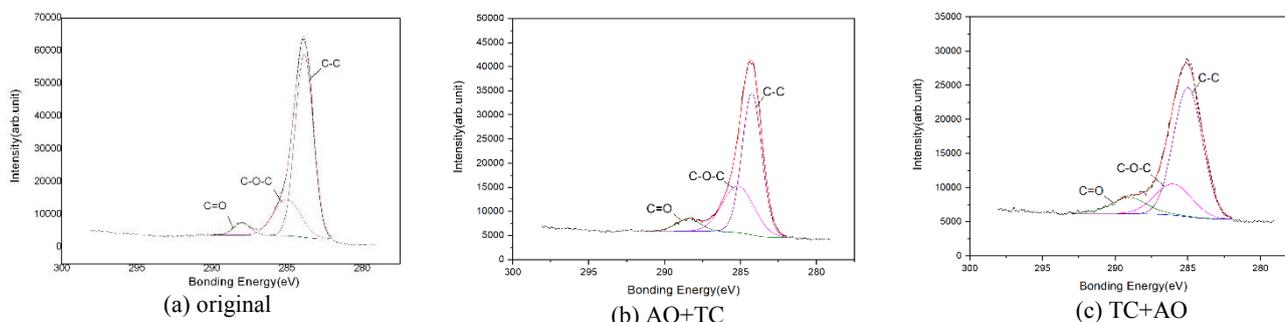


Figure 7. C1s spectrum of M40/DFA-N3 specimen surface before/after AO&TC sequent tests

Table 3. XPS analysis of M40/DFA-N3 before/after AO&TC sequent tests

Functional Groups	Bonding Energy/eV	Relative Percentage of Specific Area/ % original/(AO+TC)/(TC+AO)
C-C	285.000	73.02/61.66/67.32
C-O-C	286.500	22.12/31.96/20.71
C=O	289.000	4.85/6.37/11.96

Based on the results demonstrated above, from the angle of surface component analysis, different test sequence generally gives rise to different effects.

4. Conclusions

In this work, composites marked as M40/DFA-1 and M40/DFA-N3 were selected to be the test material to discover the effects of sequent tests focusing on atomic oxygen (1.5×10^{20} atoms/cm²) and thermal cycling (200 cycles within $-150^\circ\text{C} \sim +150^\circ\text{C}$), especially the difference in between. Methods such as optical and micro morphology observation, inter-laminar strength test, SEM as well as XPS were introduced to obtain and understand the data and phenomenon. Basically, we conclude that:

- No obvious difference could be identified between different sequences in the optical observation;
- In the SEM examination, it is distinguishing that the erosion under AO+TC sequence is much severe than that under TC+AO sequence;
- Atomic oxygen could weaken the inter-laminar shear strength, while thermal cycling could improve it. Given the condition in this work, for both materials it is consistent that, different test sequence led to different changes in inter-laminar strengths even to the different direction. In which, AO+TC reduced it whereas TC+AO raised it, which is out of our expectation. To discuss the reason, it might be the extend TC brought to the improvement in strength is larger than the extend AO reduced in it. Furtherly, and most critical in the point, post-curing procedure which fastened the crosslinking combination, thus pre-improved the resistance to atomic oxygen in the sequence of TC+AO.
- According to the result from XPS analysis, for both materials, more C-C bonds were preserved in the TC+AO test. Given the recognition that when oxygen atom breaks in a C-C bond to become a C-O-C bond, it would then lower the resin's crosslinking density, so the result from XPS demonstrates more C-C bonds retained in TC+AO test than that in AO+TC could result in the enhancement in mechanical property. That is matching with the inter-laminar strength measurements.

In conclusion, limited by the ground-based facilities, space environment tests incorporating atomic oxygen and thermal cycling, couldn't be conducted simultaneously so far, bidirectional sequent tests are employed instead. However, results from the sequent tests indicate that, different sequences lead to different results. Thus, we can deduce that, synergistic effects of space environment do not equals to the rough summation of single factor effects, which might be between the effects from the different sequent tests. To discover mechanisms beneath the appearance even to a much broader scope, a large amount of work needs to be conquered in the future.

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