

A COMPARISON OF ATOMIC OXYGEN DEGRADATION IN LOW EARTH ORBIT AND IN A PLASMA ETCHER

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ABSTRACT

In low Earth orbit (LEO) significant degradation of certain materials occurs from exposure to atomic oxygen (AO). Orbital opportunities to study this degradation for specific materials are limited and expensive. While plasma etchers are commonly used in ground-based studies because of their low cost and convenience, the environment produced in an etcher chamber differs greatly from the LEO environment. Because of the differences in environment, the validity of using etcher data has remained an open question. In this paper, degradation data for 22 materials from the orbital experiment Evaluation of Oxygen Interaction with Materials (EOIM-III) are compared with data from EOIM-III control specimens exposed in a typical plasma etcher. This comparison indicates that, when carefully considered, plasma etcher results can produce order-of-magnitude estimates of orbital degradation. This allows the etcher to be used to screen unacceptable materials from further, more expensive tests.

INTRODUCTION

When the early shuttle missions returned from low Earth orbit (LEO), it was discovered that certain materials were damaged. The surface morphology and chemical composition of many exposed materials changed, and some paints and coatings eroded away completely. Researchers determined that atomic oxygen (AO), which exists in LEO, had degraded these materials. This degradation has the potential to compromise the performance and lifetime of missions with significant time in LEO. Orbital studies of AO degradation are limited by opportunity, expense, and difficulty in monitoring specimens. Over the last decade, significant progress has been made in producing an environment on the ground that simulates the LEO environment.

Early in the search for ground AO simulations, researchers turned to commercially available plasma etchers because of their low cost and convenience. However, the environment produced in a plasma etcher is very different from the environment in LEO. These differences raise the question of whether the data collected in an etcher can be used in any way to determine how a material will behave in orbit. This paper addresses that question.

The Materials Engineering Branch at Goddard Space Flight Center (GSFC) participated in Evaluation of Oxygen Interaction with Materials (EOIM-III), an orbital experiment that flew aboard the shuttle Atlantis STS-46 in July 1992. Thirty-two of the 82 specimens that flew were submitted and evaluated upon return by the Materials Engineering Branch. Participation in this experiment provided the Branch with a unique opportunity to explore the relationship between orbital and etcher results. In April 1995, the previously unexposed control specimens for these thirty-two materials were tested in an SPI PlasmaPrep X, parallel-plate plasma etcher.

The results of the orbital and etcher exposures were analyzed and compared, and the appropriate use for etcher results was defined.

BACKGROUND

AO Degradation in LEO and Plasma Etcher

A spacecraft traveling at an altitude between 200 and 600 km has a velocity of roughly 8 km/sec. At this altitude, ultraviolet radiation separates the normally diatomic oxygen molecule into two highly reactive oxygen atoms. Although the density and proportions vary, for an average LEO (~275 km) mission the atmosphere is approximately 80% AO. The high velocity of the spacecraft yields an average AO impact energy for ram-oriented surfaces of 5 eV. This energy is sufficient to break chemical bonds and oxidize materials. Many materials have volatile oxidation products which, in LEO, results in mass loss and potential contamination problems for the rest of the spacecraft. Often there are also changes to the thermal and specular properties of the material.

A linear etcher, like the PlasmaPrep X, can be described as a large, parallel plate capacitor enclosed in a vacuum chamber. Low pressure molecular oxygen is fed into the etcher chamber. As with a capacitor, opposite charges are placed on two parallel plates, setting up an electromagnetic field between them. When the molecules enter the field, they become polarized and are attracted to one of the plates. The charges on the plates are quickly reversed, so that the molecule becomes attracted to the opposite plate. The charges are reversed at 13.56 MHz. This is so fast that the resulting vibration of the oxygen separates the molecule and excites the gas into a plasma.

By nature, a plasma consists of many different species. In addition to atomic oxygen, the etcher chamber has molecular, ionic and anionic oxygen at various excited states. There is also a significant density of free electrons. As these species excite and de-excite they emit UV radiation. When the AO collides with a specimen in the etcher chamber the average impact energy is only 0.05 eV.

Table 1 summarizes the differences between the average LEO (ram-oriented) and average etcher environments. Given all these differences, it seems quite reasonable to question whether etcher degradation rates are at all useful in predicting orbital degradation.

Table 1: Comparison of Orbital and Etcher Environments (ref. 1)

Property	(units)	Etcher	LEO (~200 km)
Oxygen Atom Flux	(atoms/cm ² ·sec)	10 ¹⁹ - 10 ²⁰	10 ¹⁵
Oxygen Atom Energy	(eV)	0.04 - 0.06	5
Oxygen Molecule Flux	(mol/cm ² ·sec)	10 ²¹	10 ¹³
Oxygen Molecule Energy	(eV)	0.05	10
Electron Density	(cm ⁻³)	10 ¹⁰	10 ¹⁵
Electron Energy	(eV)	5	0.1
UV Flux	(cm ⁻² ·sec ⁻¹)	10 ¹³	4 x 10 ¹¹
UV Wavelength	(nm)	at 130	at 121.6

Reported Results

Orbital degradation is reported as the reaction efficiency (R_e). R_e is defined as:

$$R_e (\text{cm}^3/\text{atom}) = \frac{\Delta V}{\# \text{ O atoms}} \quad (1)$$

where ΔV is the volume-loss and # O atoms is the number of oxygen atoms that collided with the specimen.

The volume loss is usually found from measured mass-loss (Δm_s) and the known density of the material (ρ_s). Since atomic oxygen degradation is a surface interaction, the mass-loss is normalized to mass-loss-per-unit-area. Thus, equation 1 becomes

$$R_v (\text{cm}^3/\text{atom}) = \frac{\Delta m_s}{A_s \cdot \rho_s \cdot F} \quad (2)$$

where A_s is the exposed area of the specimen, and the fluence (F) is the number of atoms that collided with each square centimeter of the specimen.

There are several ways to determine the number of oxygen atoms to which a specimen was exposed. One method is to measure the mass loss of a control specimen of Kapton that was exposed to the environment along with the test specimen. The R_v of Kapton has been determined in numerous orbital experiments, and it has proven consistent enough that the mass- or volume-loss-per-unit-area of Kapton can be used to estimate the fluence (F) of exposures. Rearranging terms from (1) and (2) yields:

$$F (\text{atoms}/\text{cm}^2) = \frac{\Delta V_{ko}}{A_k \cdot R_{ek}} \quad (3)$$

where ΔV_{ko} is the volume-loss of Kapton in orbit, A_k is the exposed surface area of the Kapton, and R_{ek} is the reaction efficiency of Kapton ($3.0 \times 10^{-24} \text{ cm}^3/\text{atom}$, see ref. 5).

There are no adequate models or instruments to measure the fluence inside an etcher directly. Instead, the known R_{ek} and the control specimen volume-loss following an etcher exposure (ΔV_{ke}) are used to calculate the etcher effective fluence (F_f).

$$F_f (\text{atoms}/\text{cm}^2) = \frac{\Delta V_{ke}}{A_k \cdot R_{ek}} \quad (4)$$

F_f is not the actual AO fluence in the etcher chamber. It is defined as the *orbital* fluence that would cause the volume loss the Kapton control specimen experienced in the etcher experiment.

Finally, the F_f is used to calculate the reaction efficiency of the specimen material (R_{es}).

$$R_{es} (\text{cm}^3/\text{atom}) = \frac{\Delta V_s}{A_s \cdot F_f} \quad (5)$$

R_{es} is defined as the volume-loss-per-unit-area of the test specimen, ($\Delta V_s/A_s$), divided by the effective fluence (F_f from equation 4). Again, the volume-loss of the material is usually determined from the mass-loss (Δm_s) and the known density (ρ_s) of the material. Thus, equation 5 becomes

$$R_v (\text{cm}^3/\text{atom}) = \frac{\Delta m_s}{A_s \cdot \rho_s \cdot F_f} \quad (6)$$

EXPERIMENTAL

Evaluation of Oxygen Interaction with Materials (EOIM-III)

Most of the specimens used in the EOIM-III orbital experiment were layered. Each material specimen was placed on an aluminum substrate disk with a layer of M-69 polyester film, double-sided with acrylic adhesive. Many of the material specimens consisted of a material (e.g., Teflon) with a

protective coating (e.g., SiO₂). During the orbital experiment, an aluminum plate with standardized openings (specimen cover) held the flight coupons to prevent degradation along the sides of the specimens (see Figure 1). Therefore, the top layer was considered to be the test material for each specimen.

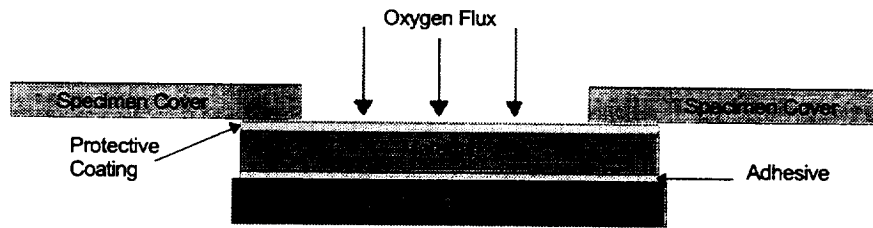


Figure 1
Illustration of EOIM-III specimen layers (ref. 2)

Six coupons were assembled for each specimen material. The flight coupons and the five control coupons were subjected to identical conditions until the flight coupons were tested in orbit. Prior to flight, all the specimens were vacuum baked and then weighed (ref. 2).

The exposed area of the flight coupons was 0.8107 cm², dictated by the size of the openings in the specimen cover. They were exposed for 58 hours, for a total AO fluence of 2.07×10^{20} atoms/cm² (ref. 2). Following orbital exposure, the flight samples were exposed to vacuum at room temperature, to remove any contaminants that may have accumulated during re-entry or transportation back to Goddard, and then weighed again. Since the density of the exposed surface was unavailable for most specimens, the reaction efficiencies were reported in g/atom (equation 7).

$$R_s (\text{g/atom}) = \frac{\Delta m_s}{A_s \cdot \text{fluence}} \quad (7)$$

In addition to the mass measurements, many other tests were performed before and after the orbital exposure. These tests included surface analysis using an M-Probe ESCA to determine elemental composition and chemical bonding states in the first 100 Å of the surface of each specimen (ref. 2).

Plasma Etcher Tests

The Materials Engineering Branch tested one complete set of the EOIM-III control specimens in a PlasmaPrep X, parallel-plate plasma etcher. The samples were removed from the sealed packages and placed in a desiccator for one month. Then, one at a time, the specimens were tested according to the following procedure.

1. Test specimen was analyzed in the ESCA
2. Test specimen and Kapton control specimen were weighed on a Mettler digital balance.
3. Test specimen and Kapton control specimen were exposed simultaneously in the etcher for 5 hours with the following settings:
 - O₂ feed gas 0.3 mbar;
 - forward power 120 watts, reflected power 10 to 14 watts;
 - average effective fluence (from Kapton erosion) = $2.5 \pm 0.7 \times 10^{20}$ atom/cm².
4. Test and Kapton control specimens were re-weighed.
5. Test specimen was analyzed in the ESCA and changes in surface composition were noted.
6. Mass loss and reaction efficiency were calculated (equation 7).

This procedure was completed for each of the 32 specimens. During etching, all specimens rested on an alumina plate to minimize charging or shielding effects from contact with the lower etcher plate. A second plate of alumina with three specimen holes was placed over the specimens to standardize the exposed area and to minimize edge erosion. The area of each of the three holes was $5.067 \pm 0.008 \text{ cm}^2$.

RESULTS AND DISCUSSION

Data

From the original set of 32 specimens, 10 were removed from consideration for this paper. The reasons for their removal are discussed in detail later, and a table summarizing the data for all the specimens tested can be found in the Appendix. The following table (Table 2) summarizes the data from both the orbital (ref. 2) and etcher tests for the materials that were used for this paper. The estimated uncertainty (σ) in etcher reaction efficiency comes from propagated errors in weight and area measurements of both the test and control specimens. In Table 2, the specimens are listed in order of orbital reaction efficiency. Note that the order would be different if they were listed in order of etcher reaction efficiency.

Table 2: Orbital and Etcher results

Specimen	Material	Reaction Efficiency (g/atom)		
		Orbit Re	Etcher Re	σ
W23	CV-2500 on Silver Teflon, Ag up	1.00E-25	1.90E-25	3.3E-26
X12	CV-1144-0 on X389-7 Aluminized Beta Cloth	2.79E-25	4.29E-25	7.9E-26
SP4	G11 Epoxy Fiberglass, Selfextinguishing	5.27E-25	3.03E-25	5.3E-26
X37	SiOx on Tefzel, SiOx up	5.48E-25	2.96E-25	5.1E-26
W22	CV-1144-0 on Silver Teflon, Ag up	8.70E-25	4.24E-25	7.7E-26
X68	828/140/TiO2	8.70E-25	3.23E-24	5.6E-25
W27	CV1142	1.15E-24	2.37E-25	4.2E-26
X70	Ni coating on Llumalloy, Ni up	1.41E-24	1.44E-24	2.5E-25
X38	CV-2500 on Kapton	1.60E-24	4.33E-25	7.6E-26
W28	CV-1500 Black Silicone	1.98E-24	5.65E-25	9.9E-26
W29	CV-2566 Silicone	1.98E-24	1.15E-24	2.0E-25
SP2	CV-2500 on Delrin II 900 NC10	2.01E-24	2.23E-24	3.9E-25
W26	CV-1144-0 on Kapton	2.35E-24	5.79E-25	1.0E-25
W30	Kapton 200XC, 2 mil	3.08E-24	3.81E-24	6.7E-25
X42	TPX	4.43E-24	7.30E-24	1.3E-24
X43	Cryovac Film	4.54E-24	6.56E-24	1.1E-24
X40	Victrex PES 4800 G	4.96E-24	5.94E-24	1.0E-24
X36	PEEK 450G	5.12E-24	5.33E-24	9.3E-25
X50	3M Tape #5	5.60E-24	5.56E-24	9.8E-25
X44	Llumalloy	5.72E-24	5.50E-24	9.7E-25
X35	ULTEM 1000	6.10E-24	5.40E-24	9.4E-25
SP3	Delrin II 900 NC10	1.58E-23	1.64E-23	2.8E-24

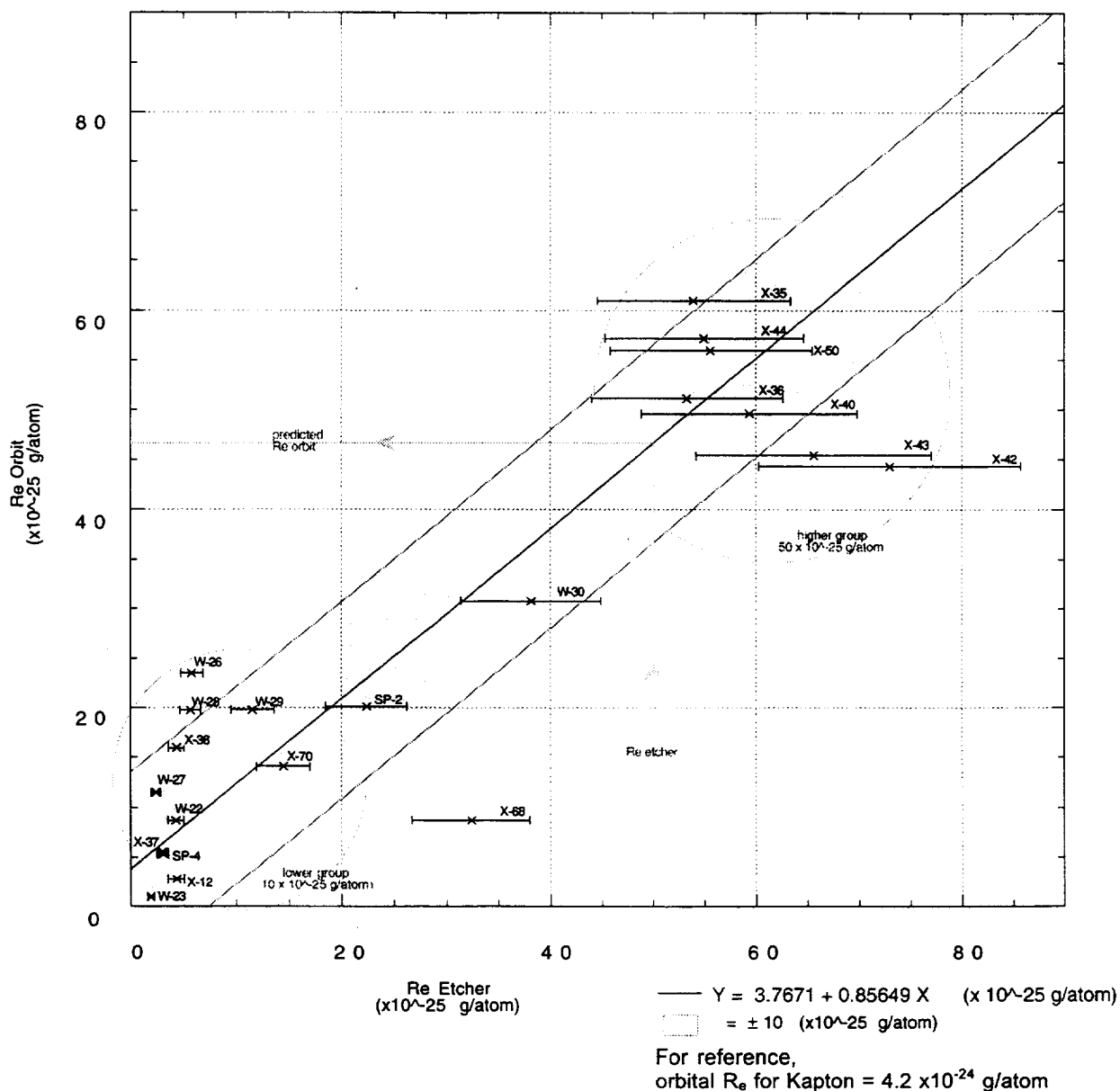
Numerical Analysis

The orbital and etcher results were compared using Figure 2. The x-axis is etcher R_e in the units 10^{-25} g/atom, and the y-axis is orbital R_e in the same units. Each data point represents one specimen, where the x-coordinate is the etcher reaction efficiency and the y-coordinate is the orbital reaction efficiency. The horizontal error bars correspond to the etcher estimated uncertainty values (σ) in Table 2. The uncertainties of the orbital reaction efficiency measurements were not reported.

Figure 2

Reaction Efficiency (Re)
Orbit vs Etcher

no fluorinated polymers, no negative orbital results



A linear regression was performed on the data, and the resulting equation is recorded at the bottom of the graph. The uncertainty of the linear regression (i.e., average uncertainty in the predicted orbital reaction efficiency) is represented on Figure 2 by the gray area surrounding the regression line. It is the average of the difference between the predicted R_{eo} and experimental R_{eo} from all the specimens.

The equation resulting from the linear regression in Figure 2, with estimated uncertainty is

$$Y = 3.7671 + 0.856 X, \pm 10 \text{ (} \times 10^{-25} \text{ g/atom)} \quad (8)$$

where Y is the predicted orbital reaction efficiency based on etcher reaction efficiency, and X is the reaction efficiency of the material in the etcher.

Ideally, equation 8 could be used to predict orbital degradation. A material would be tested in the etcher, and the resulting etcher reaction efficiency would be entered as X in equation 8. The calculation would then give a predicted orbital reaction efficiency. Unfortunately, the uncertainty is too large for this to be meaningful.

A simple and useful interpretation of the graph is that it shows data in two sets: one in the range of 50×10^{-25} g/atom in orbit, and one at roughly 10×10^{-25} g/atom in orbit. Although the line from the fit of the data does pass through these two groups, within each of these groups the trend of the data does not follow the linear regression. Therefore, equation 8 cannot be used to predict in orbit where a specimen will fall with respect to the other materials within its group.

The magnitude of the uncertainty illustrates this weakness in the linear regression. An etcher reaction efficiency can indicate in which of the two groups a specimen falls, but it can not indicate whether that material will perform better or worse than other materials within its group in orbit. For instance, in the higher group, an etcher efficiency of 50×10^{-25} g/atom yields a predicted orbital efficiency of $47 \pm 10 \times 10^{-25}$ g/atom. Given this uncertainty, it is not really reasonable to assume that a specimen with an etcher efficiency of 50×10^{-25} g/atom would indicate an orbital efficiency greater than another specimen with an etcher efficiency of 45×10^{-25} g/atom. Instead, it can only be concluded that the material is much more reactive in orbit than a material with an etcher efficiency of roughly 10×10^{-25} g/atom.

These conclusions are useful because knowing to which "grouping" a material belongs can, in some cases, limit the number of materials that need to be tested in a better, more expensive simulation. In other cases this rough estimate can eliminate completely the need for a better simulation. This allows the etcher to be used as a screening device.

An examination of the chemical composition of the two groups yields some useful information (see Table 3). Of the twelve specimens in the lower group ($R_{eo} \sim 10 \times 10^{-25}$ g/atom) nine are silicones, one is SiO_x (X-37), one is nickel sputtered (X-70), and one is an epoxy (SP-4). There is no obvious relationship between the ranking of the materials within this group and type of material. While the lower group consists mostly of silicon-containing materials, there are no silicon-containing materials in the higher group ($R_{eo} \sim 50 \times 10^{-25}$ g/atom). Instead, the higher group contains a variety of organic polymers without silicon. Of the seven specimens in the higher group, five contain oxygen in the normal (i.e., pre-exposure) polymer chain, and the remaining two (X-42, X-43) do not. It should be noted that three specimens do not fall into either of the groups. The reaction efficiencies of SP-3 (Delrin) in LEO and in the etcher were much higher than the other specimens, so it is not displayed on the graph. The other two outlier specimens are W-30 (Kapton 200XC) and X-68 (TiO_2).

Table 3: Summary of Material Classes vs. R_e Groups

Low R_e Group	High R_e Group	Outliers
9 silicones 1 SiO _x 1 epoxy 1 Ni sputter	No silicon-containing specimens 5 organic polymers w/ oxygen 2 organic polymers no oxygen	1 Delrin (high R_e) 1 Kapton 200XC 1 TiO ₂

Exclusion of Specimens

The fact that 10 out of 32 specimens had to be excluded from the study illustrates the difficulty in using the etcher even as a screening device. The reasons for removing the specimens fall into two groups: specimen configuration problems and type of material problems.

The samples X-67, X-68, X-69, SP-1, and SP-5 were eliminated due to configuration problems that made it impossible to compare etcher and orbital results. The most common problems were from protective coatings that did not cover the substrate completely. In the etcher, when the alumina specimen cover was placed over them some of the substrate was still exposed at the edge. The specimen cover in orbit had smaller openings so that no substrate was exposed. This meant that in the etcher the substrate could lose mass, but in orbit only the coating could lose mass. Thus, a comparison between etcher and orbital results could not be made.

A second set of specimens (W-24, W-25, X-11, X-31, and X-32) was excluded because their exposed surfaces contained fluorine. Historically, fluorinated materials have had inconsistent reaction efficiencies both in orbit and in simulations. For orbital results these inconsistencies are slowly being traced to synergistic effects from other orbital conditions (e.g., ultraviolet radiation). These orbital conditions are impossible to mimic with a plasma etcher, but there is more to the problem.

In many tests, etcher reaction efficiencies for fluorinated polymers have been three or four times higher than those attained in orbital tests. A recent study suggests the higher pressure and closed system of the etcher chamber as the cause (see ref. 4). When a fluorinated polymer reacts with AO, fluorine is often released by the oxidation process. In the etcher, this fluorine is excited into a plasma by the same conditions that create the oxygen plasma, and the resulting atomic fluorine degrades materials even more readily than the atomic oxygen does. In orbit, the low pressure and high impact energy carry the fluorine away before it can react significantly with the rest of the material. The conditions in the etcher allow the fluorine to remain in close contact with the material, causing degradation that would not occur in orbit. This means that in the etcher fluorinated compounds degrade much more rapidly than in orbit, and through a process different from what occurs in orbit (ref. 4). For this reason the fluorinated polymers were removed from the study.

ESCA analysis of the specimens after etcher exposure confirms this theory. On the surface of several of the fluorinated polymers some of the carbon-fluorine (-CF₂-) bonds were converted to carbon-hydrogen (-CFH-), suggesting the release of some fluorine. In addition, following the etching of a fluorinated material, fluorine was found on several subsequent specimens with fluorine concentration decreasing from one run to the next. It seems likely that the atomic fluorine reacts with the etcher chamber and remains in the etcher after the fluorinated specimen is removed. Therefore, the fluorine can be transferred to the next several test specimens.

CONCLUSIONS AND SUMMARY

For many materials, atomic oxygen degradation in low Earth orbit causes mass loss, changes in material properties, and contamination problems. Such degradation can compromise the performance and lifetime of missions with any significant duration in LEO. Plasma etchers are commonly used to

study this degradation, and the results of this paper indicate that when the results are carefully considered there is still value in such experiments.

Etcher results can be used to predict orbital reaction efficiencies with an average uncertainty of 10×10^{-25} g/atom. For some applications, this order-of-magnitude estimate is adequate. When greater precision is necessary, etchers can be used to limit the number of specimens that must be sent for better analysis (e.g., materials with predicted orbital reaction efficiencies greater than 40×10^{-25} g/atom could be excluded). Since etcher tests are significantly less expensive than better simulations, the etcher can be a valuable screening tool for predicting the performance of materials in LEO.

This study also shows that not every material is appropriate for etcher testing. Fluorinated compounds should not be tested in an etcher because evidence indicates degradation of these materials occurs through a process that is significantly different from what occurs in orbit. In addition, fluorine released during the oxidation process can contaminate the chamber and be transferred to the surface of future specimens. These problems with oxidation products may not be unique to fluorinated compounds; therefore, the likely oxidation products of every specimen should be considered before it is tested in an etcher.

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APPENDIX

The following table summarizes the data from both the orbital and etcher tests.

Specimen	Material	Reaction Efficiency (g/atom)			
		Orbit Re	Etcher Re	σ	
SP5	G-11, Non-selfextinguishing	-3.62E-24	3.32E-24	5.8E-25	
X69	X389-7 Aluminized Beta Cloth, Al up	-6.33E-26	3.61E-25	6.3E-26	
W23	CV-2500 on Silver Teflon, Ag up	1.00E-25	1.90E-25	3.3E-26	
X11	X-389-7 Aluminized Beta Cloth, Beta up	1.37E-25	3.06E-24	5.5E-25	*
W24	Silver Teflon, Teflon up	2.42E-25	8.53E-24	1.5E-24	*
X67	Uralane 5753LV A/B	2.53E-25	4.94E-24	8.6E-25	
X12	CV-1144-0 on X389-7 Aluminized Beta Cloth	2.79E-25	4.29E-25	7.9E-26	
SP4	G11 Epoxy Fiberglass, Selfextinguishing	5.27E-25	3.03E-25	5.3E-26	
X37	SiOx on Tefzel, SiOx up	5.48E-25	2.96E-25	5.1E-26	
X66	Uralane 5750LV A/B	7.80E-25	4.80E-24	8.5E-25	
W22	CV-1144-0 on Silver Teflon, Ag up	8.70E-25	4.24E-25	7.7E-26	
X68	828/140/TiO2	8.70E-25	3.23E-24	5.6E-25	
W27	CV1142	1.15E-24	2.37E-25	4.2E-26	
X70	Ni coating on Llumalloy, Ni up	1.41E-24	1.44E-24	2.5E-25	
X38	Kapton w/ CV-2500	1.60E-24	4.33E-25	7.6E-26	
X32	Tefzel 500LZ	1.74E-24	6.24E-24	1.1E-24	*
W28	CV-1500 Black Silicone	1.98E-24	5.65E-25	9.9E-26	
W29	CV-2566 Silicone	1.98E-24	1.15E-24	2.0E-25	
SP2	CV-2500 on Delrin II 900 NC10	2.01E-24	2.23E-24	3.9E-25	
W26	CV-1144-0 on Kapton	2.35E-24	5.79E-25	1.0E-25	
X31	Aclar 22C, 5 mil	2.84E-24	1.03E-23	1.8E-24	*
W30	Kapton 200XC, 2 mil	3.08E-24	3.81E-24	6.7E-25	
SP1	CV-1144-0 on Delrin II900 NC10	3.85E-24	6.58E-24	1.2E-24	
X42	TPX	4.43E-24	7.30E-24	1.3E-24	
X43	Cryovac Film	4.54E-24	6.56E-24	1.1E-24	
X40	Victrex PES 4800 G	4.96E-24	5.94E-24	1.0E-24	
X36	PEEK 450G	5.12E-24	5.33E-24	9.3E-25	
X50	3M Tape #5	5.60E-24	5.56E-24	9.8E-25	
X44	Llumalloy	5.72E-24	5.50E-24	9.7E-25	
X35	ULTEM 1000	6.10E-24	5.40E-24	9.4E-25	
W25	Silver Teflon w/hole	1.05E-23	3.16E-25	5.6E-26	*
SP3	Delrin II 900 NC10	1.58E-23	1.64E-23	2.8E-24	

The specimens in bold were removed from consideration for the paper.

* fluorinated compound